

Sudan University of Science & Technology

College of Graduate Studies



Study of Electrical, Magnetic and OpticalProperties of some Seeds and their PH and E- Coli Counts when Changing Nano Size and Solvents در اسة الخواص الكهربية والمغناطيسية والضوئية لبعض البذور والأس الهيدروجيني وبكتيريا الإشريشية القولونية عند تغيير الحجم النانوي والمذيبات

A thesis Submitted in Fulfillment of the Requirementsfor the Degree of Doctor of Philosophy in Physics.

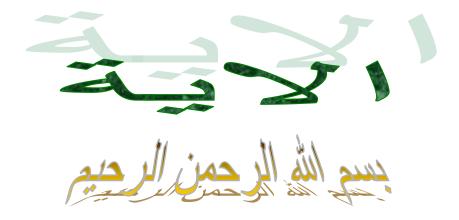
By:

Elshafia Abd-Alla Ali Elzeen

Supervisor

Prof. Mubarak Dirar Abd-Alla Yagoub

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صدق الله العظيم

# **Dedication**

# First, I dedicate this work

# To everyone who loves me and pray for

Me

## In this world

# To my great

# Father

# My supportive brothers and sisters

# My beautiful children

# My friends

# And special Dedication to my beloved

# Wife

# And every person who helped me to finish this

# Research

# Acknowledgement

At first all thanks due to almighty Allah who gives me the power to go forward in a way illuminated with his merciful guidance. My thanks to prof. <u>Mubarak Dirar Abd-alla</u> supervisor who introduced me to this research and helped me through out this project, and special thanks for <u>Dr. Abd-Alsakhi Suleiman Mohammed.</u>

Secondly many thanks are extended to and my colleagues and to all people who made it possible for me accomplish this achievement.

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### Abstract

Seeds are widely used for human food and food supplements. Water is mainly used as its host environment. This requires studying their properties separately and this work stems from the importance of seeds in human life. Therefore this research aimed to study the physical properties of some widely used seeds to see how their nano structure and energy bonds affect their physical properties. To do this experimental and analytical method were used. Four seeds samples which are azadirchta indica (neem), trigonella foenum graecum (fenugreek), negella sativa, and elettaria cardamomum were grinded in a powder form and their nano structure were examined using x-ray diffraction (XRD) spectrometer. The results show that the powders are nano particles having dimensions of 100, 27.3, 11.1, and 48.4nm respectively. Their energy bonds were determined using fourier transform form infrared spectrometer (FTIR). The FTIR results shows characteristic O-H,& Hbond, Fe2o3& Fe2o4, C-Br & N-O, and C-F & -C=C-H-H energy bonds. These four seeds were dissolved in water, methanol and ethanol solvents respectively to form 12 samples. The optical absorption, electric permittivity and conductivity were studied using ultraviolet spectrometer (UV). The results obtained shows that the electric permittivity, conductivity, magnetic permeability changes with the energy bonds, solvents and nano size.

The PH results indicate change of it with solvent types and time, where it decreases for ethanol, methanol and water. It also decreases with time.

#### المستخلص

تستخدم البذور على نطاق واسع في أغذية الإنسان والمكملات الغذائية و تستخدم المياه بشكل رئيسي كبيئة مضيفة لها ، وهذا يتطلب دراسة خصائصها بشكل منفصل وهذا العمل ينبع من أهمية البذور في حياة الإنسان . لذا هدف هذا البحث دراسة الخواص الفيزيائية لبعض البذور المستخدمه على نطاق واسع لمعرفة كيف تؤثر بنيتها الناتوية وروابط الطاقة على خصائصها الفيزيائية . للقيام بذلك أستخدمت الطريقة التحريبية والتحليلية تم سحن أربع عينات من البذور وهي (بذور النيم وبذور الحلية والحبة السوداء التحريبية والتحليلية تم سحن أربع عينات من البذور وهي (بذور النيم وبذور الحلية والحبة السوداء التحريبية والتحليلية تم سحن أربع عينات من البذور وهي (بذور النيم وبذور الحلية والحبة السوداء (الكمون) ، وبذور الهبهان ) في شكل مسحوق وتم فحص هيكلها الناتوي بإستخدام مطيافية حيود الاشعة السينية (الكمون) ، وبذور الهبهان ) في شكل مسحوق وتم فحص هيكلها الناتوي بإستخدام مطيافية حيود الاشعة السينية (الكمون) ، وبذور الهبهان ) في شكل مسحوق وتم فحص هيكلها الناتوي بإستخدام مطيافية حيود الاشعة السينية (الكمون) ، وبذور الهبهان ) في شكل مسحوق وتم فحص هيكلها الناتوي باستخدام مطيافية حيود الاشعة السينية (الكمون) ، وبذور الهبهان ) في شكل مسحوق وتم فحص هيكلها الناتوي باستخدام مطيافية حيود الاشعة السينية (الكمون) ، وبذور الهبهان ) في شكل مسحوق وتم فحص هيكلها الناتوي باستخدام مطيافية حيود الاشعة السينية (الكمون) ، وبذور الهبهان ) في شكل مسحوق وتم فحص هيكلها الناتوي باستخدام مطيافية حيود الائمية ألماسينية (الكمون) ، وبذور الهبهان ) في شكل مسحوق وتم فحص هيكلها الناتوية بأبعاد (100 ، 7.2 ، 10.1 والموليف الائمية تحت الحمراء (FTIR)، روابط الطاقة المميزة الحاصة بهم باستخدام تحويل فورير من مطياف الائشعة تحت الحمراء ((U)، الحالية المميزة المالاصة والاربعة في الماء والإيثانول على الائشعة تحت الحمراء (الموالي لتشكل 12 عينة . تما دراسة الإمتصاص البصري ، السماحية والموسيلية الكهربية باستخدام التوالي لتشكل 12 عينة . تما دراسة الإمتصاص البصري ، السماحية والموصيلية الكهربائية ، والموصلية التوالي لتشكل 21 عينة . ورابط الطاقة ، والمانوى مولي ملوى يوالموصلية الكهربية ، والموصلية ماليسية التوالي ملي ألول مالمو موق البنفسجي ((U)) أظهرت الناني وحمم النام . وحمم اليا ألول والالية ، والموصلية الموصلية الم

و تشير نتائج الأس الهيدروجيني إلى تغيرها مع انواع المذيبات والزمن ، حيث تنخفض نسبة الإيثانول والميانول والمياء ، كما انه يتناقص مع مرور الوقت .

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# Chapter one INTRODUCTION

#### **1.1. Background to Nanotechnology**

Nanotechnology is an interdisciplinary field of the science of physics, chemistry and materials sciences, meant for the design and fabrication of nano materials and their applications. This branch of science is destined to understand the fundamental physical and chemical properties and the phenomenon of nano materials and nanostructures and because of the novel applications of nano materials, the science of nano materials has evolved as a frontline research area [1, 2]. Feynman pointed out the importance of nanotechnology at the annual meeting of the American Physical Society in 1959, in the classic science lecture entitled "There is plenty of room at bottom". In the last three decades, many discoveries and inventions have been made in the field of nano science in terms of fabricating new materials and utilizing them for applications. Various new experimental techniques, with unique and desired properties of nano materials fabrication have been discovered.

Considerable research efforts with different synthesis methods have given birth to different class of nano materials, typically classified into four categories based on their dimensionality: Zero dimensional (0D), one dimensional (1D), two dimensional (2D), and three dimensional (3D). Quantum dots and the individual molecules fall in the 0-dimensional structures where the nanoparticles are isolated from each other and the electrons are confined in 3-D [3, 5]. One dimensional nanostructure (nano wires, nano rods, nano tubes, and nano obelte) where at least one of the dimensions goes in the range of nano scale order are being highly utilized and have versatile application in the nano device fabrications. Thin nano

films lie in the two dimensional structures and are studied extensively for the utilization of nano device application [6, 7].

Three – dimensional nano materials include powders, fibrous, multilayer and polyc rystalline materials in which the 0D, 1D and 2D structural elements are in close contact with each other and from interfaces. An important type of three-dimensional nano structured materials is a compact or consolidated (balk) polycrystalline with nano size grains, whose entire volume is filled with those nano grains. Applications of nano science in the field of medicine specifically that are related to the food supplements and seeds used in human food are very important. This comes from the fact that recently the food supplements and seeds were shown to play an important role in human health and life.

### **1.2. Research problem**

No intensive work was done for explaining magnetic, electrical and thermodynamic properties of nano matter. Namely the food supplements seeds.

#### **1.3.** Research significance

The importance of the study emerged from the wide applications of nano materials. Its importance is also come from expected results that can be used to synthesize nano materials with new properties. The production of such material is extremely useful in industrial development and applications.

## 1.4. Objective

#### **1.4.1.** General objective

To determine the optical and structural properties of azadirchta indica (neem seeds), trigonella foenum graecum, negella sativa and elettaria cardamomum. Were dissolved in three different solvents (water, methanol and ethanol) made by (sol gel and chemo thermal) methods.

## **1.4.2.Specific objectives**

- To prepare azadirchta indica (neem seeds), trigonella foenum graecum (fenugreek), negella sativa and elettaria cardamomum, by being dissolved in three different solvents (water, methanol and ethanol) by (sol gel and chemo thermal) methods.
- To study optical properties and electrical properties as well as chemical bonds using ultraviolet and fourier transform infrared spectrometers.
- To determine the structure parameter by using x-ray diffraction device.

### 1.5. Thesis layout

This thesis consists of five chapters. Chapter one is the introduction. Chapter two is the theoretical background and previous studies. Chapter three is for materials and methods, while chapter four is concerned with results. Chapter five is devoted for discussion, conclusion, and recommendation.

## **CHAPTER TWO**

### THEORETICAL BACKGROUND AND PERVIOUS STUDIES

#### 2.1. Introduction:

This chapter represents of information about the theoretical background of researches of nanoparticels, syntheses of nanoparticels, Atomic structure, spectroscopy, optical properties, bacteria and previous studies.

#### 2.2. Nanotechnology :

Nano is Greek word(meaning dwarf) refers to a reduction of size, or time, by  $10^{-9}$  which is one thousand times smaller than a micron. One nanometer (nm) is one billionth of a meter and it is also equivalent to ten Angstroms [10].

**Nano - science** is a new discipline concerned with the unique properties associated with nano materials, which are assemblies of atoms or molecules on a nano scale. Nano science is actually the study of object or particles and its phenomena at a very small scale, ranging roughly from 1 to 100 nm. (Nano) refers to a scale of size in the metric system.

**Nano** – **object:** Material confine in on, two, or three dimension at the nano scale this includes nanoparticles (all three dimension in the nano scale), nano fiber (two dimensions in the nano scale), nano fiber are further divided into nano tubes (hollow nano fiber) nano rods (solid nano fiber) and nano wire (electrically conducting or semiconducting nano fiber). However, the term nano-object is not very popular.

**Particle:** It is a minute piece of matter with defines physical boundaries. A particle can move as a unite. This general particles definition applies to nano- objects.

**Nanoparticles:** Are the nano- objects all external dimensions are in the nano scale. Nanoparticles can have amorphous or crystalline form and their surfaces can act carriers for liquid droplets or gases.

**Nano - particular matter:** It refers to a collection of nanoparticles, emphasizing their collective behavior.

**Agglomerate:** It is a group held together by weak forces such as van deer Waal's forces, some electrostatic forces, and surface tension. It should be noted that agglomerate will usually retain a high surface- to- volume ratio.

**Aggregate:** It is a group of particles held together by strong forces such as those associated with covalent or metallic bonds. It should be noted that an aggregate may retain a high surface- to- volume ratio.

**Nanotechnology:** is the construction and use of functional structures designed from atomic or molecular scale with at least one characteristic dimension measured in nanometer. Their size allows them to exhibit novel and significantly improved physical, chemical, and biological properties, phenomena, and processes because of their size. Thus, nanotechnology can be define as research and development that involves measuring and manipulating matter at the atomic, molecular, and super a molecular levels at scales measured in approximately 1-100 nm in at least one dimension. When characteristic structural features are intermediate between isolated atoms and balk materials in the range of approximately 1-100nm, the objects often display physical attributes different from those displayed by either atoms or bulk materials. The term "nanotechnology " is by and large used as a reference for both Nano science and nanotechnology especially in the public domain. We should distinguish between Nano science and nanotechnology.

Nano science is a convergence of physics, chemistry, materials science, and biology, which deals with the manipulation and characterization of matter on scales between the molecular and the micron size. Nanotechnology is an emerging engineering discipline that applies methods from Nano science to create products [11].

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**Nano-coatings** Applications of nano-coatings are used to stabilize highly explosive materials, making it much safer to handle nuclear and other warheads.

**Nano-coatings** can also stabilize biological and chemical agents, making them longer lasting and diversifying their means of delivery. Radio –frequency shield coatings could provide privacy and security to shield buildings and wireless networks form ratio waves.

**Nano-optics** Nano-engineered negative index met materials are moving stealth technology toward cloaking and invisibility, based on their ability to deflect light away from around an object rather than reflect it. Optical fibers married with nano wires portend the advent of solar-rechargeable, portable and wearable electronic devices [12].

#### **2.3.** Simple Lattices

Although no semiconductors crystallize into simple lattice they form the basis for understanding the more complicated semiconductors structure. We well use them to illustrate some of the more important concepts involved in forming a mathematical description of the crystal lattice.

A concept most useful in specifying the underlying geometry of crystal structure it has the property that arrangement of lattice sites around any given lattice sits is the same as that around any other mathematically, A Bravais Lattice consist of all poivrts generated by the vectors.

There is no unique way to choose  $a_i$ . we choose  $a_1$  as shortest period of the lattice ,  $a_2$  as the shortest period not parallel to  $a_1$ ,  $a_3$  as the shortest period not coplanar to  $a_1$  and  $a_2$ . The  $a_i$  which generate Bravais lattice is known as vectors.

In the simple cubic structure, which has an atom at each corner of a cubic of dimension the Bravais Lattice can be determined by three mutually orthogonal vectors.

 $a_1 = a_x$ ,  $a_2 = a_y$ ,  $a_3 = a_z$ 

Where x, y and z are cartesian unit vectors. This set of vectors demonstrates the basic symmetry of the structure and it is easy to see that entire Bravais Lattice can be constructed with these vectors this set of primitive vectors is not unique, however, in defining the simple cubic Brava's can also be used to construct the Lattice as well as an infinite number of other sets.

The body centered cubic structure has an atom at each corner of cubic dimension and one at the point determined by the intersection of the cubic body diagonals, another lattice of interest in semiconductor crystal structure is the hexagonal close packed lattice. Although not a bravais lattice, because the lattice sites are not equivalent it consist of two interpenetrating simple hexagonal lattice which are bravais lattice. The simple hexagonal lattice consists of lattice site at each corn of an equilateral triangle of side, with an additional set of points on triangle at a distance above the first [13].

#### 2.4. Crystal structure

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Most of solid materials possess crystalline structure that means spatial periodicity or translation symmetry. All the lattice can be obtained by repetition of a building block called basis. We assume that there are 3 non-coplanar vectors  $a_1$ ;  $a_2$ , and  $a_3$  that leave all the properties of the crystal unchanged after the shift as a whole by any of those vectors. As a result, any lattice point  $R_0$  could be obtained from another point R as

$$\mathbf{R}' = \mathbf{R} + \mathbf{m}_1 \, \mathbf{a}_1 + \mathbf{m}_2 \, \mathbf{a}_2 + \mathbf{m}_3 \, \mathbf{a}_3 \tag{2.2}$$

Where  $m_1$ ,  $m_2$  and  $m_3$  are integers. Such a lattice of building blocks is called the Bravais lattice. The crystal structure could be understood by the combination of the propertied of the building block (basis) and of the Bravais lattice. The natural way to describe a crystal structure is a set of point group operations which involve operations applied around a point of the lattice. We shall see that symmetry provide important restrictions upon vibration and electron properties. We will concentrate on cubic lattices which are very important for many materials [14].

The crystal structure is now produced by attaching the basis to each of these lattice points. (lattice +basis =crystal structure), there is no unique way to choose the primitive vectors for a given lattice, and often the choice depends upon convenience. The volume cell enclosed by the primitive vectors is called the primitive unite cell. Because of the periodicity of a lattice, it is useful to define the symmetry of the structure. The symmetry is defined via a set of point group operation which involves a set of operations applied around a point. The operations involve rotation, reflection, and inversion. The symmetry plays a very important role in the electronic properties of the crystals [15]. The size of the unit cell and the arrangement of atoms in a crystal may be determined from measurement of the x-rays by the crystal.

### 2.5. X-Ray Diffraction and Bragg's Law

X-ray diffract meters consist of three basic elements: an X-ray tube, a sample holder, and X-ray detector. X-ray is generated in a cathode ray tube by heating a filament to produce, accelerating the electrons toward a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to dislodge inner shell electrons of the target material, characteristic X-ray spectra are produced. These spectra consist of several components, the most common being  $K_{\alpha}$  and  $K_{\beta}$ .  $K_{\alpha}$  Consists, in part, of  $K_{\alpha_1}$  and

 $K_{\alpha_2}$ .  $K_{\alpha_1}$  Has a slightly shorter wavelength and twice the intensity as  $K_{\alpha_2}$ . The specific wavelengths are characteristic of the target material (Cu, Fe, Mo and Cr). Filtering, by foils or crystal monochrometers, is required to produce monochromatic X-rays needed. For diffraction  $K_{\alpha_1}$  and  $K_{\alpha_2}$  are sufficiently close in wavelength such that a weighted average of the two is used. Copper is the most common target material for single-crystal diffraction, with  $CuK_{\alpha}$  radiation =1.5418A<sup>0</sup>. These X-ray are collimated and direction on to the sample. As the sample and detector are rotated, the intensity of the reflected X-rays recorded. When the geometry of the incident X-rays impinging the sample, as the sample and detector are rotated, the intensity of the reflected X-rays is recorded. When the geometry of the incident X-rays impinge the sample satisfies the Bragg Equation, constructive interference occurs and a peak in intensity occurs. A detector records and processes this X-ray signal and converts the signal to a count rate which is then output to a device such as a printer or computer monitor. The geometry of an X-ray diffract to meter is such that the sample rotates in the path of the collimated X-ray beam at an angle  $\theta$  while the X-ray detector is mounted on an arm to collect the diffracted X-rays and rotates at an angle of  $2\theta$ . The instrument used to maintain the angle and rotate the sample is termed a goniometry [16].

Figure (2.1) illustrates how diffraction of x-rays by crystal planes allows one to derive lattice spacing's by using the Bragg's law. X-ray techniques provide one of the non-destructive methods of measuring residual stresses. X-ray stress (strain) analysis is based on the Bragg diffraction equation:

$$n\lambda = 2d\sin\theta$$
 (2-3)

Where (n) is an integer (diffraction order),  $\lambda$  the wavelength of the incident X-ray, d the interplanar spacing of the polycrystalline material under consideration, and  $\Theta$  is the Bragg angle

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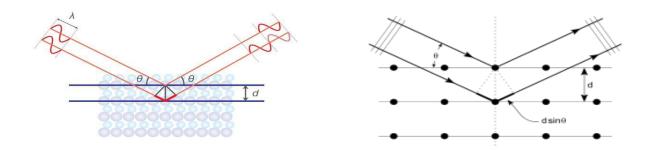


Figure (2.1) illustrates how diffraction of X-rays by crystal planes allows one to derive lattice spacing's by using the Bragg's Law.

#### 2.6. Millar indices

Used to specify directions and planes, the directions and planes could be in lattices or in crystals. This scheme advantage of eliminating all fractions from the notation for a plane. In the hexagonal system, which has four crystallographic axis, it is group of three number that indicates the orientation of a plane or set of parallel planes of atoms in a crystal, If each atoms in the crystal is represented by a point and these points are connected by lines, the resulting lattice may be divided into a number of identical blocks, or unit cells the intersecting edges of one of the unit cells defines a set of crystallographic axis, and the Millar indices are determined by the intersection of the plane with these axis. The reciprocals of these intercepts are computed, and fractions are cleared to give the three Millar indices (h k l) [17].

#### 2.7. Bravais Lattice

These are several ways to describe a lattice, the most fundamental description is known as the bravais lattice. In words, a bravais Lattice is an array of discrete points with an arrangement and orientation that look exactly the same from one another. Thus, a bravais lattice can refer to one of the 14 different types of unit cells that a crystal structure can be made up.

### 2.8. Type of Bravais Lattice

Out of 14 type of Bravais Lattice some seven type of Bravais Lattices in three- dimensional, the Letters a ,b and c have been used to denote the dimensions of the unit cells where as the Letters  $\alpha,\beta$  and  $\gamma$  denote the corresponding angle in the unit[17].



### 2.8.1.Cubic system

In bravais lattice with cubic systems, the following relationships can be observed  $\mathbf{a}=\mathbf{b}=\mathbf{c}=90^{\circ}$   $\alpha=\beta=\gamma=90^{\circ}$ 

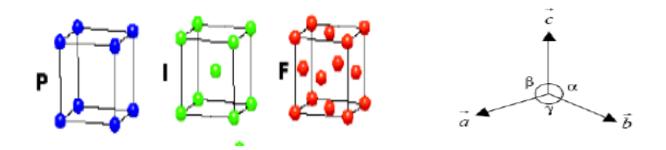


Figure (2.2): The illustrated three possible types of cubic cells.

### 2.8.2.Orthorhombic system

In Bravais Lattice with Orthorhombic systems obey the following equation

$$\mathbf{a} \neq \mathbf{b} \neq \mathbf{c}$$

$$\alpha = \beta = \gamma = 90^{\circ}$$

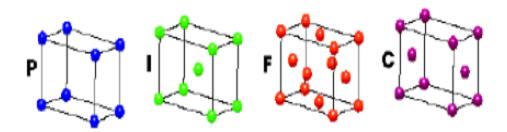


Figure (2-3): the four types of Orthorhombic systems.

## 2.8.3.Tetragonal system

In tetragonal Bravais lattice, the following relations are observed  $\mathbf{a}=\mathbf{b}\neq\mathbf{c}$ 

 $\alpha = \beta = \gamma = 90^{\circ}$ 

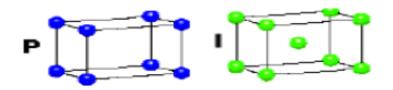


Figure (2.4): the two types of tetragonal systems

### 2.8.4.Monoclinic system

Bravais lattice having monoclinic systems obeys the following relations:  $\mathbf{a} \neq \mathbf{b} \neq \mathbf{c}$ 

 $\beta = \gamma = 90^{\circ} \text{ and } \alpha \neq 90^{\circ}$ 

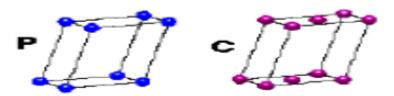


Figure (2.5) the two possible types of monoclinic systems.

## 2.8.5.Triclinic System

There exists only one type of triclinic bravais lattice, which is a primitive cell. It obeys the following relations:  $\mathbf{a} \neq \mathbf{b} \neq \mathbf{c}$  and  $\alpha \neq \beta \neq \gamma \neq 90^{\circ}$ 

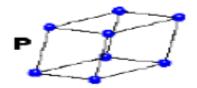


Figure (2,6) simple type of triclinic cell

### 2.8.6.Trigonal system

Only the primitive unit cell for a trigonal system exists. Its cell relation is given by:  $a=b=c=90^{\circ} \quad \alpha=\beta=\gamma \neq 90^{\circ}$ 

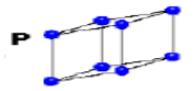


Figure (2.7): type of primitive trigonal cell.

### 2.8.7.Hexagonal system

The only type of hexagonal bravais lattice is the simple hexagonal cell. It has following relation between cell side angles  $\mathbf{a} = \mathbf{b} \neq \mathbf{c}$  and  $\alpha = \beta = 90^{\circ}$  and  $\gamma = 120^{\circ}$ 

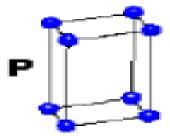


Figure (2.8): simple type of hexagonal cell.

#### 2.9. Fourier Transform Infrared Spectroscopy(FTIR)

Infrared (IR) spectroscopy is one-photon effect and the photon absorption results in a vibrational motion of a molecule. Infrared spectra originate from the vibrational motions of atoms in chemical bounds within the molecular structure. When a beam of light containing the (IR) radiation interacts with a sample [19]. Fourier Spectroscopy is a general term that describes the analysis of any varying signal into its constituent frequency components, Fourier Transport Infrared spectroscopy (FTIR) is a reliable method of Infrared (IR) spectroscopy and offers several analytical opportunities in academic, analytical and forensic laps, and FT-IR spectroscopy includes the absorption. Reflection, emission, or photo acoustic spectrum obtained by Fourier transform of an optical interferogram [20].

The infrared regions  $(10 - 1400 \text{ cm}^{-1})$  of the electromagnetic spectrum is divided into three regions: the near-, mid-, and far- IR. The mid-IR (400-4000cm<sup>-1</sup>) is the most commonly used region for analysis as all molecular vibrations in this characteristic absorbance frequencies and primary molecular vibrations in this range. Mid- infrared spectroscopy, Methods are based on studying the interaction of infrared radiation with samples. As IR radiation is passed through a sample, specific wavelengths are absorbed causing the chemical bonds in the material to undergo vibrations such as stretching, contracting, and bending. Functional groups present in a molecule tend to absorb IR radiation in the same wave number. Range regardless of other structures in the molecule, and spectral peaks are derived from the absorption of bond vibrational energy changes in the IR region. Thus there is a correlation between IR band positions and chemical structural groups, IR spectra can provide qualitative information, such as the concentration of bacteria in a growth medium. An IR radiation is measured by calculating the intensity of the IR radiation before and after it passes through a sample and the spectrum is traditionally plotted with Y- axis units as absorbance or transmittance and of X-

axis as wave number units. For quantitative purposes it is necessary to plot the spectrum in absorbance units [21].

FT-IR absorbance spectra follow Beer's law, which relates concentration to absorbance as in equation (2.4)

$$A_{\lambda} = L_{\lambda}C \tag{2.4}$$

Where  $A_{\lambda} \equiv$  Absorbance.  $L_{\lambda} \equiv$  Path length, C  $\equiv$  concentration.

Transmittance is not directly to the concentration and is defined in Equation (2.5)

$$T = \frac{I_S}{I_R} \%$$
(2.5)

Where  $I_S \equiv$  Intensity of  $I_R$  beam after passing through the sample,  $I_R \equiv$  intensity of IR beam before passing through the sample,  $T \equiv$  Transmittance.

#### 2.10. Preparation of Nanoparticles

Synthesis of nano metal and metal oxide materials involves substantial synthetic ingenuity. Although one can develop a balanced method to the synthesis of nano materials, there is continually an element of serendipity. A verity of nonmaterial's has been synthesized in the last several decades by the old-style ceramic method, which includes mixing and heating them at elevated temperature with transitional grinding when necessary. Some of the important chemical methods of synthesis of oxides are co precipitation and precursor methods, ion exchange and sol-gel technique, top chemical methods, hydrothermal / solvothermal approaches, combustion technique (self- propagating high – and low- temperature method and solution combustion method), microwave- assisted and liquid interface method, etc. some of them have been briefly described in the following sections [22]. Synthesis of nano particles based on two approaches namely, top- down approach and bottom-up, based on this several methods have been developed.

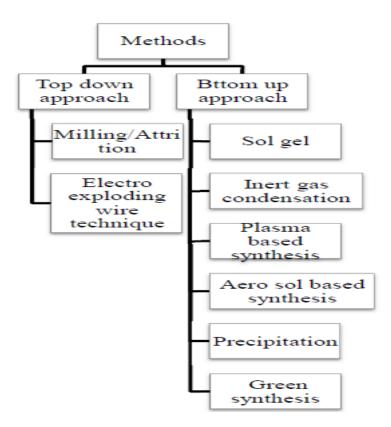


Figure (2.9): methods of nanoparticles synthesis

### 2.10.1. Top down approach:

It involves breaking down of large size bulk material into nano size particles, could be done by milling, attrition process and electro explosion wire technique. It is a quick manufacturing process but requires more energy, so it is not suitable for large scale production. Another drawback of top down approach is imperfections of surface structure such defects have a significant impact on the physical and other properties of nanoparticles [23]. For example Cu nanoparticles synthesized by the top down approach of electro exploding wire (EEW) technique [24]. In this technique copper plate is kept inside suitable medium such as water, current approximately 1010 A/m2 is applied to the medium through the copper wire which leads to melting and evaporation of copper metal plate taken place. Evaporation of metal creates the plasma which readily dispersed in the media followed

centrifugation to separate the particles. Similarly [25] synthesized the silver Nanoparticles and analysis the structural properties such as XRD, SEM, and UV -Vis spectroscopy. (2-4) nm size of iron particle produced with agglomerate structure using high energy ball mill under room temperature followed by particle separation using sieve shaker and the structure morphology was confirmed by high resolution electron micro scope (HREM) [26]. They also postulated that high degree of deformation of material occur in mechanical milling process due to repeated impact between the ball and particles. In Conventional milling process the heat generation will be more as well as the particle agglomeration also happened, in order to avoid this wet milling method had been developed by [27]. It is a popular method because of its applicability and suitability can be applied for all kind of materials. Optimize the parameters such as milling time, rotational velocity of agitator shaft, filling ratio of grinding media, flow velocity of circulation system for preparation of ZnO Nanoparticles using Taguchi method, Response Surface method and genetic algorithm approach [28].

#### 2.10.2. **Bottom up approach**

Bottom approach refers to building of material from molecule by molecule, atom by atom and cluster by cluster. During the assembling process physical forces acting on the nano structure used to combine the particles in to a larger one. For synthesis of complex nano structures, nano technologist mostly prefer bottom up approach because the advantage of this approach is to precise control of particle size resulting good optical, electronic and other properties [29]. The common methods involved in bottom up approach as follows:

#### **2.10.2.1.** Sol – gel methods

The sol gel technique is well established technique for synthesis of colloidal nanoparticles from liquid phase. The main advantages of sol gel technique are versality, low temperature process and flexible rheology allowing easy shaping. sol gel process is well suitable for synthesis of oxide nanoparticles and composite nano powder. The most commonly used precursors are alkyl oxides due to their commercial availability and high accountability of MOR bond allowing eloquent tailoring in situ during processing. The procedure involving sol gel technique as follows [30],

- Preparation of homogeneous solution by dissociation of metal organic precursor in the organic solvent or in organic salt solution.
- Transformation of precursor oxide in to a highly cross linked solid.
- Hydrolysis leads to sol, dispersion of colloidal particle in liquid done by suitable reagents (generally water).
- Further condensation leads to gel, a rigid inter connected organic network.

For example  $TiO_2$  based Nanoparticles synthesis is one of the hot topic for the nanotechnologist because of its wide range of applications in photo catalysis, shape memory alloy and solar cells [31].

#### 2.10.2.2. Co Precipitation

Co precipitation is a typical wet chemical process, also called as solvent displacement method. It was widely used method, due to simplicity, economic and reproducibility. Main constituents are need to prepare the nano particle are polymer phase can be synthetic or natural, polymer solvent usually ethanol, acetone, hexane and non solvent polymer. Nanoparticles are created by a rapid diffusion of polymer solvent into a non solvent polymer phase by mixing the polymer solution finally. Interfacial tension between two phases creates a maximum surface area could leads to spontaneous precipitation of nanoparticles. Flow sheet for co-precipitation technique are summarized below, Anti microbial activity of CuO nanoparticles synthesized by precipitation technique using copper acetate and sodium hydroxide as a precursors and reducing agent respectively. Characterization of particles carried out by XRD, TEM and EDS analysis. XRD reveals that Nanoparticles are monoclinic crystal similarly EDS confirmed that absence of impurities in the prepared copper oxide nano particles [32]. Nano fluids are new variety of fluids in which nano size material such as nanoparticles, nano tube, nano fibers, nano wire, and nano rods in a base [33] The same author produced two types of zinc oxide (ZnO) nanoparticles, presence and absence of capping agents for analyzing its behavior in Nan fluids. The experiment was conducted by utilizing zinc nitrate, sodium hydroxide, starch and poly vinyl 2pyrroledone which act as precursors, reducing agents, stabilizers and capping agents respectively. The following conclusion was made by researchers, the capped ZnO nanoparticles were unagglomerate and had regular define structure than uncapped nanoparticles. Magnetite nano particles play a vital role in many areas such as magnetic drug target, magnetic resonance imaging for clinical diagnosis, recording material and catalyst. Co precipitation of magnetic  $Fe_3O_4$  nano particle had the successful rate up to 99.7 [34].

### 2.10.2.3. Inert Gas Condensation

The inert gas condensation (IGC) technique, in which Nanoparticles are formed via the evaporation of a metallic source in an inert gas, had been extensively used to produce fine nano particles. Perhaps this method is well suited for manufacturing metal nano particles, since metals are vaporized at reasonable rate at attainable temperature. Process for making of copper metal nanoparticles are summarized below,

- Metal is vaporized inside the chamber, into which an inert gas typically argon or helium or neon is periodically admitted.
- Once the atom is boil off immediately loss its energy, by colliding of vaporized atom with inert gas.
- The vapor cooled by liquid nitrogen, to form nanoparticles in the range of 2-100nm.

Bimetallic Au/Pd Nanoparticles synthesized by highly size controlled inert gas condensation technique [35]. The resultant Nanoparticles were analyzed by TEM, mass spectroscopy, electron microscopy, atomic force microscopy, and XRD to verify the geometry and distribution of metals [36]. Done a research on formation of Cu and Si Nano clusters by a sputtering or inert gas (Ar) aggregation type cluster source. The size of cluster was controlled by the operating parameters such as sputtering pressure and gas-flow rate. The actual mean cluster size was compared with theoretical mean cluster size. The end result illustrated that the peak cluster size was decreased with increased Ar gas flow rate, due to the time spent by the cluster within the agglomeration region decreases resulting in lower size.

#### 2.10.2.4. Green synthesis

Nano particles are produced by the physical and chemical method need a longer time for synthesis to overcome this kind of problem, recent development of Nano technology is green synthesis which use of biological system like plants and micro organism [37]. More over green synthesis of nano particle is an environmentally friendly method without any harmful and expensive chemicals [38]. Synthesis of metal nano particles using plant extracts are considered as a cost effective and hence used as an economically viable method for large scale production [39]. The mechanism behind the formation of nano particles explained by several researchers, they postulated that bio reduction of nano particles owing to numerous bio molecules (vitamins, amino acids, proteins, phenolic acids, alkaloids, etc) in the plant and micro organisms [37]. Phenolic acids are considered as a powerful anti oxidants, possess hydroxyl and carboxyl groups that are able to bind metals. The active hydrogen may responsible for reduction of metal ions into formation of metal nanoparticles [40]. The overview of green synthesis is easily understand by the Fig (2.10), when precursor is added to the leaf extract it changes the color which indicate the formation of Nano catalyst.

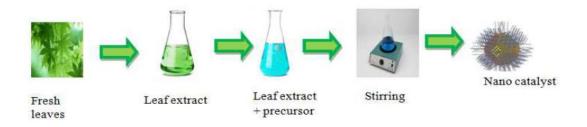


Figure (2.10) illustrates Green synthesis of nano catalyst

Averrhoa bilimbi fruit extract broth synthesized gold and silver nano particles using the salt chloroauric acid and silver nitrate respectively. Color change to yellow and violet indicated that formation of silver and gold nano particles. The rate of formation of nano particles were slow at lower concentration of salt thus lowers the absorbance in UV spectrometer. They also suggested that alcohols, amines and phenols in the averrhoa bilimbi fruit might cause reduction and capping of Nanoparticles [41]. Another investigation demonstrated for ZnO nano particles from Calotropis Gigantea leaf extract [42]. The synthesized particles were characterized by SEM determined the spherical shape of Nano particles with a size of 10-15 nm and XRD study also revealed the size of the Nano particle. It was offered that rate of synthesis related to reaction temperature, high temperature allow faster growth rate. More over increasing temperature leads to small size of nano particles. Stabilization of nano particles using microwave heating is an emerging field of nano particle synthesis. Gold nano particles synthesized from Hibiscus rosa - sinensis leaves and also optimizing the parameters like time and micro wave power have been performed [43]. Finally they concluded that higher microwave power at lower time produce Nanoparticles at faster rate because of rapid uniform heating. Anti microbial activity against multiple drug resistant bacteria by silver Nanoparticles were produced from jamun leaf broth by utilizing silver nitrate as a precursor studied [44].

#### 2.11. Spectroscopy

In principle, a spectrometer is the simplest of scientific instruments. The term spectroscope derives from two root words: the Latin word spectrum, meaning image, and the Greek word skopein, to view, (e.g. microscope, telescope, etc.).

So a spectroscope is an instrument that permits visual observation of spectra. Instruments that record a spectral image on a photographic plate (the spectroscope plus the tube plate holder) are commonly called spectrographs. A spectroscope can thus "fingerprint" a material by disclosing what elements the material contains and in what proportions. In certain cases it is not even necessary to touch the object being studied. Almost anything that emits, absorbs, or reflects light Not only can elements be identified (the method is called spectrochemical or elemental analysis), but information can also be obtained on the constituents of the elements – the electrons and atomic nuclei – as well as the atoms and molecules them - selves. This aspect is sometimes referred to as atomic or molecular spectroscopy. Spectroscopy has been the means where by physicists and chemists have learned most of what they now know about the nature of matter. It was originally limited to

visible light, but new ways of generating and detecting other kinds of energy are constantly being developed [45]. There are many types of spectroscopy and they are used to detect, identify and quantify data about material samples as gases, liquids and solid. As such, spectroscopy is used to determine both the chemical composition as well as measure the physical properties of material. UV- Vis spectroscopy is an analytical technique that measures the amount of discrete wavelengths of UV or Visible light that are absorbed by or transmitted through a sample in comparison to a reference or blank sample. This property is influenced by the sample composition, potentially providing information on what is in the sample and at what concentration. Since this spectroscopy technique relies on the use of light, let's first consider the proportional to its wavelength. Thus, shorter wavelengths of light carry more energy and longer wavelengths carry less energy. A specific amount of energy is needed to promote electrons in a substance to a higher energy state which we can detect as absorption. Electrons in different bonding environments in a substance require a different specific amount of energy to promote the electrons to a higher energy state. This is why the absorption of light occurs for different wavelengths in different substances. Humans are able to see a spectrum of visible light, from approximately 380 nm, which we see as violet, to 780nm, which we see as red. UV light has wavelengths shorter than that of visible light to approximately 100nm. Therefore, light can be described by its wavelength, which can be useful in UV-Vis spectroscopy to analyze or identify different substances by locating the specific wavelengths corresponding to maximum absorbance [46].

#### **2.11.1.Ultraviolet** – visible spectroscopy

Ultraviolet and visible spectroscopy is absorption spectroscopy uses electromagnetic radiations between 190nm to 800 nm and is divided into the

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ultraviolet (UV, 190 -400 nm) and visible (VIS, 400-800 nm) regions. Since the absorption of ultraviolet or visible radiation by a molecule leads transition among electronic energy levels of the molecule, it is also often called as processes can including reflection, scattering, absorbance. fluorescence occur, or phosphorescence (absorption and reemission), and photochemical reaction (absorbance and bond breaking). In general, when measuring UV- visible spectra, we want only absorbance to occur. Because light is a form of energy, absorption of by matter causes the energy content of the molecules (or atoms) to increase. The total potential energy of a molecule generally is represented as the sum of its electronic, vibration, and rotational energies [47].

In the UV-Vis spectral range transitions between electronic energy levels can be observed, which determine the absorption bands in the UV-Vis region. An electron is excited when the frequency of the incident electromagnetic radiation is the same as the difference of energy between two electronic states. This difference of energy depends on the electronic structure of the molecule and of its "environment". For a transition to happen after absorption of radiation it is necessary to have a dislocation of charge and some rules [48].

## **2.11.2. Optical properties**

Optical properties are very useful for the quantitive determination of the electronic band structure of martial. In this study optical absorbance, reflectivity, transmission, and refraction provide the way to determine the dielectric constant of the normal human blood sample, which is related to the band structure. The dielectric constant is related to the optical conductivity. The term ''optical conductivity '' means the electrical conductivity in the presence of an alternating electric field [49].

### 2.11.2.1. Absorption

The intensity of the net absorbed radiation is dependent on the character of the medium as well as the path length within. The intensity of transmitted or non - absorbed radiation continuously decreases with distance (x) that the light traverses:

$$I_T = I_0 e^{-\beta x} \tag{2-5}$$

Where  $I_T$  is the intensity of the non-reflected incident radiation and  $\beta$  the absorption coefficient (in mm<sup>-1</sup>), is characteristic of the particular material; furthermore, varies with wavelength of the incident radiation. The distance parameter (x) is measured from the incident surface into the material. Materials that have large values are considered highly absorptive [50].

#### 2.11.2.2. Transmission

The phenomena of absorption, reflection, and transmission may be applied to the passing of light through a transparent solid. For an incident beam of intensity ( $I_0$ ) that impinges on the front surface of a specimen of thickness (1) and absorption coefficient, the transmitted intensity at the back face  $I_T$  is

$$I_T = I_0 (1 - R)^2 e^{-\beta I}$$
(2-6)

Where (R) is the reflectance; for this expression, it is assumed that the same medium exists outside both front and back faces. Thus, the fraction of incident light that is transmitted through a transparent material depends on the losses that are incurred by absorption and reflection. Again, the sum of the reflectivity R, absorptivity (A), and transmissivity (T), is unity according to Equation (2-2). Also, each of the variables R, A, and T depends on light wavelength. This is demonstrated the transmission [49, 50].

## 2.11.2.3. Reflection

When light radiation passes from one medium into another having a different index of refraction, some of the light is scattered at the interface between the two media even if both are transparent. The reflectivity R represents the fraction of the incident light that is reflected at the interface, or

$$R = \frac{I_R}{I_0} \tag{2-7}$$

Where  $I_0$  and  $I_R$  are the intensities of the incident and reflected beams, respectively. If the light is normal (or perpendicular) to the interface,

then

$$R = \left(\frac{n_2 - n_1}{n_2 + n_1}\right)^2 \tag{2-8}$$

Where  $n_1$  and  $n_2$  are the indices of refraction of the tow media. If the incident light is not normal to the interface, R will depend on the angle of incidence. When light is transmitted from a vacuum or air into a solid, then

$$R = \left(\frac{n_2 - 1}{n_2 + 1}\right)^2 \tag{2-9}$$

Because the index of refraction of air is very nearly unity. Thus, the higher the index of refraction of the solid, the greater the reflectivity [51, 52 and 49].

#### 2.11.2.4. Absorption coefficients

Much of the information about the properties of materials is obtained when they interact with electromagnetic radiation. When a beam of light (photons) is incident on a material, the intensity is expressed by the Lambert-Beer- Law as Equation (2-4). If this condition for absorption is met, it appears that the optical intensity of the light wave. (I), is exponentially reduced while traveling through the film. If the power that is coupled into the film is denoted by  $I_0$ , gives the transmitted intensity that leaves the film of thickness d. ( $\alpha$ ) is called "absorption coefficient". From Equation (2-9) it follows that:

$$\alpha = -\frac{1}{d} \operatorname{Lin}(\frac{I}{I_0})$$

$$\alpha = \frac{1}{t} \ln[\frac{(1-R)^2}{T}]$$
(2-10)<sub>a</sub>

Where (t) is the sample thickness, (T) and (R) are transmission and reflection. But if you don't have T and R and you have Absorbance, Then:

$$\alpha = 2.303 \frac{A}{t} \tag{2-11}$$

Where  $\alpha$  is absorption coefficient, (A) is absorbance. From Beer Lambert Law:

$$I = I_0 \exp(\alpha \times t) \tag{2-12}$$

Thus,

$$\operatorname{Lin}\left(\frac{I}{I_0}\right) = \alpha \times t$$
$$\alpha = (2.303 \times A)/t$$

Where  $I_0 \equiv$  incident intensity,  $\alpha \equiv$  absorption coefficient, t = Thickness of the material. The transparent substrate has a thickness several orders of magnitude larger than (d) and has index of refraction (n) and absorption coefficient ( $\alpha = 0$ ). The index of refraction for air is taken to be  $n_0 = 1$ . In the transparent region ( $\alpha = 0$ ) the transmission is determined by (n) and (s) through multiple reflections. In the region of weak absorption  $\alpha$  is small and the transmission begins to decrease. In the medium absorption region( $\alpha$ ) is large and the transmission decreases drastically due almost exclusively to the influence of ( $\alpha$ ). If the thickness (d) is uniform, interference effects give rise to the spectrum [51, 53 and 54].

#### **2.11.2.5. Extinction coefficient**

Exctinction coefficient measure of how strongly a substance absorbs light at a particular wavelength. It is related to the absorption coefficient by:

$$k = \frac{\alpha \lambda_0}{4\pi} \tag{2-13}$$

Where  $\alpha$  is the absorption coefficient, k is the extinction coefficient, and  $\lambda_0$  is the wavelength in vacuum,  $\lambda$  is the wavelength. If ( $\lambda$ ) is in nm, multiply by 10<sup>7</sup> to get the absorption coefficient in the, units of cm<sup>-1</sup> [51].

## 2.11.2.6. Refractive index

Light that is transmitted into the interior of transparent materials experiences a decrease in velocity, and, as a result, is bent at the interface; this phenomenon is termed refraction (n) of a material is defined as the ratio of the velocity in a vacuum (c) to the velocity in the medium or :

$$n = \frac{c}{v} \tag{2-14}$$

The magnitude of (n)(or the degree of bending) will depend on the wavelength of the light. This effect is graphically demonstrated by the familiar dispersion or separation of a beam of white light into its component colors by a glass prism. Each color is deflected by a different amount as it passes into and out of the glass, which results in the optical path of light, but also, as explained shortly, it influences the fraction of incident light that is reflected at the surface. Just as Equation (2-14) defines the magnitude of (c) an equivalent expression gives the velocity of light in a medium as:

$$v = \frac{1}{\sqrt{c\mu}} \tag{2-15}$$

Where v and are respectively, the permittivity and permeability of the particular substance. From Equation (2-15), we have

$$n = \frac{c}{v} = \frac{\sqrt{\epsilon_{\mu}}}{\sqrt{\epsilon_{0\,\mu_{0}}}} = \sqrt{\epsilon_{r}\mu_{r}}$$
(2-16)

Where  $\epsilon_r$  and  $\mu_r$  are the dielectric constant and the relative magnetic permeability. Because most substances are only slightly magnetic, and

$$n \cong \sqrt{\epsilon_r} \tag{2-17}$$

Thus, for transparent materials, there is a relation between the index of refraction and the dielectric constant [54].

## 2.12. Conductivity

The electric current flow through conductors when an electric field is applied or when a potential difference is produced between the conductor terminals. The ability of the current to easily is called conductivity [55, 56]. Thus the conductivity is directly proportional to the flowing electric current (I). If the applied voltage is (V), the resistance R obeys the relation:

$$V=RI = \frac{\rho L}{A} I \qquad (2.18)$$

With  $\rho$ , *A*, *L* standing for resistivity, cross sectional area and the conductor length respectively. The electric conductivity is defined by

$$\sigma = \frac{1}{\rho} = \frac{L}{RL} \tag{2.19}$$

## 2.12.1. Electrical conductivity

The conductivity which results electric fluid and generate direct current this direct current results from applying direct potential difference between the conductor terminals. This voltage source generates direct current which flow only in one direction. The generated current may be constant or variable [57]. The electric conductivity is given according to equation (2.19) by:

$$\sigma = \frac{L}{RL} = \frac{LI}{VA} \tag{2.20}$$

## **2.12.2. Optical conductivity**

Applying oscillating voltage generates alternating current which changes its direction periodically. This alternating current can also be generated when electromagnetic waves or even light interact and enter the term optical conductivity is used to describe this current flow type. In its simplest case the current (I) is given in terms of current density (J), electron speed (v) and electron concentration (n), in the form:

$$I = JA = nevA \tag{2.21}$$

Under the electric field (E) action the electron equation of motion is given by:

$$m\frac{d\nu}{dt} = i\omega m\nu = eE \tag{2.22}$$

There form:

$$I = \frac{ne^2 AE}{i\omega m} \tag{2.23}$$

Thus equations (2.20) and (2.23) give:

$$\sigma = \frac{LI}{VA} = \frac{ne^2 AEL}{i\omega m ELA} = \frac{ne^2}{i\omega m}$$
(2.24)

This means that optical conductivity is frequency dependent.

## 2.13. Electric permittivity:

Electric field and lines of force are generated by electric charges which may be positive or negative. The ability of any material to enable electric lines of force to penetrate is called electric permittivity ( $\varepsilon$ ). This permittivity is defined in terms of the electric flux density (D) and the electric field intensity E according to the relation:

$$D = \varepsilon E \tag{2.25}$$

## 2.14. Magnetic permeability

Magnetic lines of force are generated by a magnetic bar which consists of north and south poles. The magnetic lines of force flow from the north to the south Pole. The number of lines of force penetrating normally surface unit area is called the magnetic flux density B. The magnetic force on a unit magnetic charge is called the magnetic field intensity (or strength) H. The magnetic permeability  $\mu$  is defined to satisfy

$$B = \mu H \tag{2.26}$$

## 2.15. Bacteria

All bacteria, both pathogenic and saprophytic are organisms that reproduce by binary fission. Most bacteria are capable of independent metabolic existence and growth, but species of Chlamydia and Rickettsia are obligately intracellular organisms. Bacteria cells are extremely small and are most conveniently measured in microns (10-6) m. they range in size from large cells such as Bacillus anthraces (1.0 to 1.3  $\mu m \times 3$  to 10  $\mu m$  to very small cells such as pasteurella tularensis (0.2 × 0.2 to 0.7  $\mu m$ ) Mycoplasmas (atypical pneumonia group) are even smaller, measuring 0.1 to 0.2  $\mu m$  in diameter. Bacteria therefore have a surface – tovolume ratio that is very high: about 100,000. Bacteria have characteristic shapes. The common microscopic morphologies are cocci (round or ellipsoidal cells, such as staphylococcus aureus or Streptococcus respectively); rods, such as Bacillus and clostridium species; loge, filamentous branched cells, such as actinomyces species; and comma-shaped and spiral cells, such as vibrio cholera and treponema pallidum, respectively.

**Escherichia coli** is a common member of the normal flora of the large intestine. As long as these bacteria do not acquire genetic elements encoding for virulence factors, they remain commensals. Strains that acquire bacteriophage or

plasmid DNA encoding enterotoxins or invasion factors become virulent and can cause either a plain, watery diarrhea or an inflammatory dysentery. These diseases are most familiar to westerners as traveler's diarrhea, but they are also major health problems in endemic countries, particularly among infants. Three groups of E coli are associated with diarrheal diseases. Escherichia coli strains that produce enterotoxins are called enterotoxigenic E coli (ETEC). There are numerous types of enterotoxin. Some of these toxins are cytotoxin, damaging the mucosal cells, whereas others are merely cytotoxin, inducing, only the secretion of water and electrolytes. A second group of E coli strains have invasion factors and cause tissue destruction and inflammation resembling the effects of Shigella (EIEC). A third group of serotypes, called entropathogenic E coli (EPEC), are associated with outbreaks of diarrhea in newborn nurseries, but produce no recognizable toxins or invasion factors[58].

## 2.16. Literature Review:

## **2.16.1.** Green Synthesis and Characterization of Silver Nanoparticels and Evaluation of their Antibacterial Activity using Elettaria Cardamom Seeds

This work done by Omprakash V<sup>1</sup>, thy study In present we have been producing metallic nanoparticles with toxic and harmful chemicals which are more dangerous to environment. Therefore there is a need to develop environment friendly synthesis techniques without using toxic chemicals. In this work a simple and green/eco-friendly/chemical free biosynthesis of silver nanoparticels using C seed extract as reducing agents was prepared. Nanoparticels were characterized by UV-vis absorbance spectroscopy, XRD and SEM measurements. The metal ions reduction occurs very rapidly, and the reduction of Ag ions will be completed within 4 hours. The synthesized silver nano particle distinct mono dispersity as they show particle size between the ranges of 30-80 nm. The experimental study

confirmed the formation of highly crystalline nanoparticles with uniform shape. Further, the antibacterial activity against the Bacillus subtilis was also evaluated for *Elettaria cardamom* seeds extract assisted silver nanoparticles and the later found to exhibit significant activity. And the conclusion of the study is, In the present investigation, a facile, environmentally benevolent green synthetic route is used for synthesis of silver nano particle. The Phytofabrication of silver nano particle by using seed extract of *E. cardamom* without involvement of any toxic chemicals. The metal ions reduction occurs very rapidly, and the reduction of Ag ions will be completed within 4 hours. The synthesized silver nano particle distinct mono dispersity as they show particle size between the ranges of 30-80 nm. Water soluble heterocyclic compounds such as flavones were mainly responsible for the reduction and stabilization of the nano particle. Assessment on the antibacterial effect of nano sized silver colloidal solution against Bacillus subtilis and Klebsiella plantcola reveals high efficacy of silver nano particle as a strong antibacterial agent. The present used seeds of E.cardamomom as a source which is easily obtainable and extensively useful in biomedical application [59].

## **2.16.2.** Biogenic synthesis of silver nanoparticles from leaf extract of Elettaria cardamomum and their antifungal activity against phytopathogens

This study done by Pragati Jamdagni, they study The current study reports biogenic synthesis of silver nanoparticles from *Elettaria cardamonum*. *Elettaria* leaf extract was used as reducing and capping agent for nanoparticle synthesis from parent solution of silver nitrate. Nanoparticle suspension was characterized mainly using UV-Visible spectroscopy. Synthesis parameters namely, time, metal ion concentration, leaf extract quantity, reaction temperature and pH are well known to affect the final product of synthesis and hence, were varied to assess optimum conditions for synthesis. Nanoparticles synthesized at optimum conditions were washed and characterized using Fourier Transform Infrared

Spectroscopy (FTIR), X-ray diffraction (XRD), Dynamic Light Scattering (DLS) and Transmission Electron Microscopy (TEM). Nanoparticles obtained were in the size range of 5-80 nm (TEM), with an average particle size of 29.96 nm as calculated using Debye - Scherrer formula and average hydrodynamic diameter of 32.12 nm (DLS). FTIR implicates plausible role of protein part of leaf extract in nanoparticle synthesis and DLS confirms monodisperse nature of the suspension. Nanoparticle suspension was found to be stable after four months of storage at room temperature without the addition of any stabilizing agents. Silver nanoparticles exhibited excellent antifungal activity against various fungal phytopathogens with minimum inhibitory concentration as low as 8 µg/mL for Aspergillus niger, making them potential antifungal agents in the field of agriculture. And the conclusion of the study is: The present study reports a simple and eco friendly approach for the synthesis of silver nanoparticles. Various reports are available for nanoparticle synthesis using *Elettaria* seeds but this is apparently the first report employing leaf extract of *Elettaria* for nanoparticle synthesis. Colour change of silver nitrate post addition of leaf extract is primary indication of nanoparticle synthesis which was further confirmed using UV- Visible spectroscopy, XRD, DLS and TEM. Irregularly spherical nanoparticles with a size range of 5-80 nm and average particle size of 29.96 nm were obtained. FTIR analysis implements the supposed role of plant proteins in the reduction and stabilization of nanoparticles. However, work still needs to be done to identify the actual causative agent. The synthesized nanoparticles showed good antifungal activity against all the tested fungal phytopathogens and could serve as potential antifungal agents [60].

## **2.16.3.** Green Biosynthesis Of Silver Nanoparticles from Elettaria Cardamomum (seeds) and Its In vitro Cytotoxic Activity

This paper done by Venugopal Krishnan<sup>\*1</sup>. The green synthesis of silver nanoparticles (AgNPs) using Elettaria cardamomum (seed) extract and its cytotoxicity activity against Hep-2 cell line were reported. The synthesized AgNPs using *Elettaria cardamomum* extract was preliminarily confirmed by UV- visible spectroscopy and it was further characterized by FT-IR. The UV-visible spectrum showed an absorption peak at 456 nm which reflects surface plasmon resonance (SPR) of AgNP. The green synthesized AgNPs exhibited a dose-dependent cytotoxicity against Human Larynx Carcinoma cancer(Hep-2) and the inhibitory concentration (IC50) were found to be 51µg/ml. Furthermore, the synthesized SNPs shows significant anticancer activity against Hep-2 cancer cell line. The results from this study also We report a simple, quick and efficient green synthesis of silver nanoparticles from the Elettaria cardamomum. The characterization with UV-vis spectroscopy and Fourier transmission infrared (FT-IR) is the preliminary evidence for the formation of nanoparticles. The synthesized silver nanoparticles showed promising anticancer activity against human pharynx cancer cell line. From the study, it can be concluded that the silver nanoparticles synthesized using Plant possess high anticancer activity against cell lines which further suggest the potential therapeutic use of these nanoparticles [61].

## **2.16.4.** Nigella sativa seeds based antibacterial composites: A sustainable technology for water cleansing - A review

This work done by Geetanjali Rathi<sup>a,1</sup>, Sharf Ilahi Siddiqui<sup>a,1</sup>, In this work, The presence of pollutants such as organic dyes and heavy metals in water has become a serious problem for aquatic environment. These pollutants that reach the human body through drinking water pose a serious threat to human life. These pollutants can be responsible for diabetes, cancer, high blood pressure, cardiovascular,

neurological arteriosclerosis, and many other serious diseases in the human body. The elimination of such water pollutants is an urgent need of the society. But the present adsorption techniques available are costly and out of reach of the masses. The biofilm formation by bacterial cells onto the solid surface is also a subject of concern. In this regard, many efforts have been made to achieve an effective adsorption technology. Adsorption technology using Nigella Sativa and its composites has gained attention because of the biofilm resistance activities of the Nigella Sativa. Owning to its unique physicochemical characteristics, antibacterial properties of Nigella Sativa based composites provide a preferred material for water treatment. This review puts in a nutshell the application of Nigella Sativa based composites for water treatment. The methods of synthesis and physio-chemical properties Nigella Sativa based materials by making use of various techniques have been discussed in depth. Pollutants removals from water have been discussed in light of various parameters that affect it. The implementing and reusing of the adsorbent are also detailed out. The comparative cost, antibacterial activity, adsorption capacities and partition coefficients have also been discussed. This review will definitely be useful for the scientific community to develop the more sustainable material for water purification with high quality. The results from this study also The green materials derived from plants such as leaves, seeds, and roots etc., have attracted a lot of attention as a bioadsorbent due to their wide availability and low cost. These materials are being used as an alternative to activated carbon and biochar which avoid chemical activation and high consumption of energy. Currently, green materials derived from plants are being utilized as carbon support for nanoparticles to generate the composite materials. These composites have gained great attention for water treatment. This review strongly recommends black cumin seeds based composites as a versatile adsorption material for water treatment. Black cumin based composites have shown the ability to remove the

pollutants rapidly and with high efficiency. These composites pose plenty of functional groups therefore; have a strong affinity for both organic and inorganic pollutants, due to electrostatic/non-electrostatic attraction and surface complexation. These composites also show good antibacterial activity, which can prove to be helpful in preventing the formation of biofilm. It can be concluded that composites based on black cumin seeds have achieved much exploration as promising adsorbents. In near future, there is a need to know more about the synergistic effect of many other nanoparticles with black cumin seeds. Further studies are needed to maintain the long-term bonding relationship of nanoparticles with carbon support. Permanent modification can increase the stability of nanoparticles on carbon support. In the near future, more efficient carbon support needs to be explored. This study also emphasizes the need of column and pilot-scale studies. It is recommended to investigate the effect of other competing ions on the adsorption of targeted pollutants on these composites [62].

## **2.16.5.** Antifungal and Antibacterial Assay by Silver Nanoparticels Synthesized from Aqueous Leaf Extract of Trigonella foenum-graecum

This work done by Azhar U. Khan<sup>1</sup>, This paper reports a safe, simple, green, nontoxic, and environment-friendly approach for the synthesis of silver nanoparticles (AgNPs) using aqueous leaf extract of Trigonella foenum-graecum at room temperature. The aqueous leaf extract was capable to act as a reducing, capping, and stabilizing agent. UV-visible spectroscopic, Fourier transform infrared spectroscopy (FT-IR), Xray diffraction (XRD), scanning electron microscope (SEM), and transmission electron microscopy (TEM) techniques were used to characterize the synthesized AgNPs. The synthesized AgNPs were found to be stable, face-centered cubic, crystalline, and spherical and in the range of 20–25 nm. The antimicrobial activities of the synthesized AgNPs were investigated against plant pathogenic fungi Alternaria alternata and plant pathogenic bacteria

Pseudomonas syringae. The assay results showed that A. alternata and P. syringae were inhibited at the 100 ppm of AgNPs in the respective medium. Antifungal assay shows the disruption of fungal mycelium at various places. Similarly, antibacterial assay shows the inhibition zones. These results confirmed that this protocol is a green, environment-friendly, and nontoxic method for the synthesis of AgNPs. Assay results confirmed that the synthesized AgNPs can be used as an effective growth inhibitor for various pathogenic microorganisms and applicable to control microbial systems. The results from this study also In this study, a simple, green, and one-pot green synthesis of stable AgNPs using aqueous leaf extract of T. Foenum graecum at room temperature was reported. The synthesis of AgNPs using T. foenum-graecum is a clean, green, inexpensive, ecofriendly, reliable, and safe method. The UV-visible spectroscopic, FT-IR, XRD, SEM, and TEM techniques confirmed the synthesis and well-dispersed nature of the AgNPs. The synthesized AgNPs showed antimicrobial activities against plant pathogenic fungi Alternaria alternata and plant pathogenic bacteria Pseudomonas syringae. Hence, the synthesized AgNPs can be used as an antimicrobial agent for food safety, improvement of agricultural production, and a range of applications in real life for the benefit of human beings [63].

# **2.16.6.** Synthesis of organic-inorganic hybrid nano flowers using Trigonella foenum-graecum seed extract and investigation of their anti-microbial activity This paper represented by Cevahir Ali Tinkaynak<sup>1</sup> in this study the Herein we report a green method for the synthesis of organic-inorganic hybrid nano flowers using a Trigonella foenum-graecum L. (TF) (Fenugreek seeds) extracts as an organic part and copper ions acting as an inorganic part. The organic-inorganic hybrid nano flowers using TF seed extract (TF-Cu<sup>2+</sup> hNFs) were characterized by SEM, XRD, EDX and FTIR. The morphology of the TF-Cu<sup>2+</sup> hNFs was quite spherical and mono disperse with ~18µm size. The TF-Cu<sup>2+</sup> hNF exhibited the

effective anti-bacterial activity against Enterococcus faecium, Enterococcus faecalis, Staphylococcus aureus, Bacillus cereus, Salmonella typhi and Escherichia coli at 1-10µg ml<sup>-1</sup> concentrations except against Pseudomonas aeruginosa and Haemophilus influenza. However, both TF-Cu<sup>2+</sup> hNFs and free TF extracts showed any antifungal activities against Candida albicans or Candida glabrata. The study revealed that TF-Cu<sup>2+</sup> hNFs could be used as a therapeutic agent for microbial infections and has the potential to overcome drug resistance. The results from this study also In summary, we reported a green synthesis method of organic-inorganic hybrid NFs by using Trigonella foenum graecum extract and Cu<sup>2+</sup> ions. The obtained NFs were well dispersed, uniform and spherical. The TF extract amount affected the size of the NFs. The pores on the NFs' surface almost completely disappeared when the TF extract amount was increased during the synthesis protocol, which is simple and economic. Our hNFs exhibited higher anti-microbial activity than TF extract against some Gram-positive and Gram-negative bacteria. Neither TF-Cu<sup>2+</sup> hNFs nor TF extract demonstrated any anticandidal activities against C. albicans or C. glabrata. Hence, the TF-Cu<sup>2+</sup> hNFs have potential applications due to their ability to overcome the drug resistance and can be used as a therapeutic agent in human health to overcome increased antibiotic resistance against pathogenic microorganisms [64].

# **2.16.7.** Green synthesis and Characterization of Silver nanoparticles from Nigella sativa and its application against UTI causing Bacteria

This work done by Priti Ranjan<sup>1</sup>, Merina Paul Das<sup>1</sup>, M. Sathish Kumar<sup>1</sup>, in this study sample of nigell sativa Synthesis of silver nanoparticles using seeds of *Nigella sativa* as reducing agent was evaluated in this study. Homogenized extract of *N. sativa* exposed to sunlight gave a better synthesis of nanoparticles when compared to other methods. Antibacterial activity of nanoparticles was studied against urinary tract infection (UTI) causing bacteria by disc diffusion method.

Further, the silver nanoparticles were characterized by UV, XRD, FTIR, SEM and EDAX. The findings suggest that silver nanoparticles from seeds of *N. sativa* may be effectively used against UTI causing bacteria. The results from this study also In conclusion, the bioreduction of silver ions using seeds of Negella sativa as reducing agent has been illustrated. From the present study, it is clear that the silver nanoparticles synthesized through the green route using seeds of *N. sativa* can inhibit the organisms causing urinary tract infection providing a significant zone of inhibition. Thus, the silver nanoparticles from seeds of *N. sativa* may be used in the management of urinary tract infection causing bacteria [65].

## **2.16.8.** Green Synthesis of Gold Nanoparticles Using Elettaria cardamomum (ELAICHI) Aqueous Extract

This work done by Monalisa Pattanayak and P.L. Nayak in this work a study of In the present study we explore the reducing and capping potential of aqueous extract from seed pod of Elaichi for synthesis of gold nanoparticles. The extract with different concentration reduced with HAuCl<sub>4</sub> aqueous solution at room temperature. The color change, pH change and UV-Visible spectroscopic analysis reveal the Surface Plasmon Resonance (SPR) of the final reaction product which confirms the reduction of Au<sup>3+</sup> ion to gold nanoparticles. XRD, Particle size analysis result represents strong reducing potential of Elaichi aqueous extract which can also be tested in the green synthesis of other metallic nanoparticles. The results from this study also the study demonstrates the rapid synthesis of gold nanoparticles with small sized and high crystalline. The reduction of the metal ions and stabilization of the gold nanoparticles is believed to occur by the proton releasing hydroxyl group [26], containing -terpineol, citronellol, borneol, transnerolidol, cis/trans-linalol oxides, -sitosterol, phytol, geraniol, stigmasterol or any other secondary metabolites and various acids present in extract. The concentration of elaichi extract and metal ions plays a crucial role for the synthesis of gold

nanoparticles of desired size with reaction conditions. The spectroscopic characterizations using UV-Vis, XRD and Particle size analysis were useful in providing the formation of nanoparticles and also to confirm their characteristic. From literature study proposed that hydroxyl and amine group containing components are responsible as an active reductant and capping agent, but further FTIR analysis can give evidance to understand the appropriate chemical and molecular interactions which could be responsible for the gold salt reduction.

# **2.16.9.** Azadirchta indica as a bio-material: Rapid synthesis of $Cr_5O_{12}$ shell nanoparticles to study its photocatalytic and antimicrobial properties

This work done by Karthiga Rajendaran <sup>a</sup>, in this work a study of A novel  $Cr_5O_{12}$ nanoparticle were prepared by reducing K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> using Azadirachta indica plant extracts as a reducing agent. The synthesized nanoparticles shows orthorhombic phases with a band gap for 1.27 eV and it is further conformed by SEM. The average dimension of the nano shell was about 56.99 nm. The FTIR spectrum explores the presence of the functional group of plant extract and Cr<sub>5</sub>O<sub>12</sub>. GC-MS of the aqueous extract shows the presence of many antioxidants in the leaf of Azadirchta Indica. The photocatalytic performance was analyzed based on the degradation of Methyl Orange (MO) dye. The rate constant k of AzI-Cr<sub>5</sub>O<sub>12</sub> is found to be 3.93  $\times 10^{-2}$  s - <sup>1</sup> and follows pseudo first-order kinetic at a catalyst dosage of 0.050 g/L with concentration of 20 mM of dye. Further, the antimicrobial activity of the nanoparticles was tested against Staphylococcus aureus, Candida albicans and Enterobacter. The results from this study of AzI- $Cr_5O_{12}$  nanoparticles have been successfully synthesized by simple co-precipitation method.  $Cr_5O_{12}$  nanoparticles were characterized by FT-IR, XRD, SEM and EDX techniques. The photocatalyst AzI-Cr<sub>5</sub>O<sub>12</sub> is successfully applied for the degradation of MO under visible light irradiation. The enhanced photocatalytic activity of AzI-Cr<sub>5</sub>O<sub>12</sub> is due to the suppression of electron-hole recombination by

leaf extract in  $Cr_5O_{12}$ . The reaction conditions are optimized and maximum photodegradation is achieved within 90 min with an AzI- $Cr_5O_{12}$  dosage of 0.050 g/L and MO concentration of 20 mM. A highest zone of inhibition was observed for Staphylococcus aureus and Enterobacter of about 40 and 35 mm respectively compared to other microorganisms [67].

## **2.16.10.** Nigella sativa seeds based antibacterial composites: A sustainable technology for water cleansing - A review

This work done by Geetanjali Rathi<sup>a,1</sup>, Sharf Ilahi Siddiqui<sup>a,1</sup>, in this work a study of The presence of pollutants such as organic dyes and heavy metals in water has become a serious problem for aquatic environment. These pollutants that reach the human body through drinking water pose a serious threat to human life. These pollutants can be responsible for diabetes, cancer, high blood pressure, cardiovascular, neurological arteriosclerosis, and many other serious diseases in the human body. The elimination of such water pollutants is an urgent need of the society. But the present adsorption techniques available are costly and out of reach of the masses. The biofilm formation by bacterial cells onto the solid surface is also a subject of concern. In this regard, many efforts have been made to achieve an effective adsorption technology. Adsorption technology using Negella Sativa and its composites has gained attention because of the biofilm resistance activities of the Nigella Sativa. Owning to its unique physicochemical characteristics, antibacterial properties of Nigella Sativa based composites provide a preferred material for water treatment. This review puts in a nutshell the application of Nigella Sativa based composites for water treatment. The methods of synthesis and physio-chemical properties Nigella Sativa based materials by making use of various techniques have been discussed in depth. Pollutants removals from water have been discussed in light of various parameters that affect it. The implementing and reusing of the adsorbent are also detailed out. The comparative cost,

antibacterial activity, adsorption capacities and partition coefficients have also been discussed. This review will definitely be useful for the scientific community to develop the more sustainable material for water purification with high quality. The results from this study The green materials derived from plants such as leaves, seeds, and roots etc., have attracted a lot of attention as a bioadsorbent due to their wide availability and low cost. These materials are being used as an alternative to activated carbon and biochar which avoid chemical activation and high consumption of energy. Currently, green materials derived from plants are being utilized as carbon support for nanoparticles to generate the composite materials. These composites have gained great attention for water treatment. This review strongly recommends black cumin seeds based composites as a versatile adsorption material for water treatment. Black cumin based composites have shown the ability to remove the pollutants rapidly and with high efficiency. These composites pose plenty of functional groups therefore; have a strong affinity for both organic and inorganic pollutants, due to electrostatic/non-electrostatic attraction and surface complexation. These composites also show good antibacterial activity, which can prove to be helpful in preventing the formation of biofilm. It can be concluded that composites based on black cumin seeds have achieved much exploration as promising adsorbents. In near future, there is a need to know more about the synergistic effect of many other nanoparticles with black cumin seeds. Further studies are needed to maintain the long-term bonding relationship of nanoparticles with carbon support. Permanent modification can increase the stability of nanoparticles on carbon support. In the near future, more efficient carbon support needs to be explored. This study also emphasizes the need of column and pilotscale studies. It is recommended to investigate the effect of other competing ions on the adsorption of targeted pollutants on these composites [68].

## **2.16.11.** Elettaria cardamomum seed mediated rapid synthesis of gold nanoparticles and its biological activities $\star$

This paper done by Anish Rajan<sup>\*</sup>, Angel Rose Rajan, Daizy Philip. In this study, Plant mediated synthesis of metal nanoparticles has drawn the attention of researchers due to their unique features and extensive application in various fields. The present report is on the unexploited environmentally benign rapid synthesis of gold nanoparticles of size 15.2nm using the aqueous extract of Elettaria cardamomum seeds. Studies on antioxidant, antibacterial and anticancer activities of the nanoparticles have been carried out. The invitro radical scavenging activity of the biogenic nanogold is found to be comparable to that of the reference. The as synthesized gold nanoparticles also exhibits antibacterial activity against a broad spectrum of bacterial pathogens. The antibacterial activity is found to be enhanced against S.aureus compared to E.coli and P.aeruginosa. Further, the obtained nanoparticles show excellent mcytotoxic activity towards HeLa cancer cell lines [69].

## **2.16.12.** Antioxidative Activity of Extracts from a Fenugreek Seeds (Trigonella foenum-graecum)

This paper represented by **S.** Birjees Bukhari\*, in this study the Spices and herbs posses. Antioxidant activity can be applied for preservation of lipid peroxidation in biological systems. Fenugreek (*Trigonella foenum-graecum*) is an important spice and aromatic crop its dried seeds having the wide application in a food, a flavoring, beverages and a medicine. Methanol, ethanol, dichloromethane, acetone, hexane and ethyl acetate crude extracts of Fenugreek were prepared by soxhelt extraction method. Extracts were investigated for there antioxidant and radical scavenging activities by different methods such as measurement of total phenolic content (TPC) by Folin-Ciocalteu method, flavonoid content, chelating activity, reducing power and1,1-diphenyl-2-picryl-hydrazyl (DPPH°)free radical scavenging activity.

All extract of the fenugreek exhibited antioxidant activity. These finding suggest that the fenugreek extract could act as a potent source of antioxidants. Results from different parameter were in agreement with one another. The conclusion of this paper is the From the present work, it could be concluded that the solvent play a vital role in the extraction of the constituents. As methanol and ethanol are highly polar among the solvent therefore they contain the high yield of phenolic as compared to the other solvents. An ethanolic extract of fenugreek seeds was examined for its antioxidant activity. The antioxidant activity could be correlated with the polyphenolic components present in the extract. The results gained by these methods provide some important factors responsible for the antioxidant potential of fenugreek seeds [70].

2.16.13. Green Synthesis of silver Nanoparticles and its optical properties

This work done by R. SARKAR, P. KUMBHAKAR\*, A. K. MITRA. In this study the Here we report for the first time, to the best of our knowledge, the synthesis of silver nanoparticles of varying sizes using parthenium leaf extract at a higher temperature of 100°C as well as at room temperature. The synthesized nanoparticles are characterized using Scanning electron microscope (SEM), X-ray diffractometer (XRD) and UV-visible spectrophotometer. The effect of the reaction time on the particle size has been reported. Also visible photoluminescence (PL) emissions from the synthesized silver nanoparticles have been recorded. Plant extract is very cost effective and eco friendly and thus can be economic and effective alternative for the large scale synthesis of silver nanoparticles. The conclusion of this paper is the Here we have reported for the first time, the synthesis of silver nanoparticles of varying sizes using *parthenium* leaf extract in the aqueous solution at a higher temperature of 100°C as well as at room temperature. The variation of particle size with the reaction temperature and reaction time has been reported. The structural characterizations of the samples

are performed using SEM and XRD analysis. The UV-visible optical absorption properties are measured and found the shift of SPR wavelengths with the average particle size of the synthesized samples. In addition visible photoluminescence emissions are observed from the synthesized silver nanoparticles. This green chemistry approach towards the synthesis of silver nanoparticles has many advantages. Plant extract is being eco friendly and very cost effective; the presented method can be economic and effective alternative for the large scale synthesis of silver nanoparticles in nanotechnology processing industries [71].

## 2.16.14. Elettaria Cardamomum seed mediated rapid synthesis of gold nanoparticles and its biological activities \*

This work done by Anish Rajan\*, in this work a study of Plant mediated synthesis of metal nanoparticles has drawn the attention of researchers due to their unique features and extensive application in various fields. The present report is on the unexploited environmentally benign rapid synthesis of gold nanoparticles of size 15.2 nm using the aqueous extract of Elettaria Cardamomum seeds. Studies on antioxidant, antibacterial and anticancer activities of the nanoparticles have been carried out. The invitro radical scavenging activity of the biogenic nanogold is found to be comparable to that of the reference. The as synthesized gold nanoparticle also exhibits antibacterial activity against a broad spectrum of bacterial pathogens. The antibacterial activity is found to be enhanced against S.aureus compared to E.coli and P.aeruginosa. Further, the obtained nanoparticles show excellent cytotoxic activity towards HeLa cancer cell lines [72].

## 2.16.15. Synthesis of chitosan incorporated neem seed extract (Azadirachta indica) for medical textiles

This paper represented by T.Revathi, S.Thambidurai\*. In this study in present study, eco-friendly biosynthesis of Chitosan-Neem seed (CS-NS) composite was prepared by co-precipitation method using aqueous neem seed extract. Cotton fabrics were treated with two different crosslinking agents (Glutaraldehyde and Citric acid) then the synthesized composite coated on cotton fabric by chemical linkage between the composite and the cellulose structure. As synthesized composite materials and treated cotton fabrics were characterized by Fourier transform infrared spectroscopy for functional groups confirmation, X-ray diffraction for crystalline behavior determination, UV-Visible spectroscopy analysis for optical property and High resolution scanning electron microscopy for Surface morphological properties. The antibacterial activity of CS-NS composite coated cotton fabric and CS-NS composite coated cotton fabric with crosslinking agents were tested against the gram-positive and gram negative bacteria by agar well diffusion method. The results demonstrated that CS-NS composite with crosslinked coated cotton fabric has higher antibacterial activity than without crosslinked cotton fabric. Thus the chitosan-neem seed composite may be applied to the medical textiles. The conclusion of this paper is the The chitosan and neem seed composite was prepared by co-precipitation method. Thus synthesized materials and composite coated cotton fabric with and without crosslinking agents were characterized by FTIR, XRD, UV-Vis and HR-SEM analysis. The XRD result indicates the amorphous structure of CS-NS composite. FTIR results also confirm that CS-NS composite are well coated on the cotton fabric with cross linking agents. Based on SEM results, CS-NS composite was well coated on crosslinked cotton fabrics. The significant antibacterial activity is obtained by CS NS composite coated cotton fabric with crosslinking agents. The antibacterial

results exhibited that, the use of glutaraldehyde and citric acid markedly inhibited the growth of both bacterial pathogens. It's promising candidate for medical textiles applications [73].

## **2.16.16.** Antibacterial, anticancer and antioxidant potential of silver nanoparticles engineered using Trigonella foenum-graecum seed extract

This study done by Shivangi Goyal<sup>1</sup>, Nidhi Gupta<sup>1</sup>, In this study, the authors report a simple and eco-friendly method for the synthesis of silver nanoparticles (AgNPs) using Trigonella foenum-graecum (TFG) seed extract. They explored several parameters dictating the biosynthesis of TFGAgNPs such as reaction time, temperature, concentration of AgNO<sub>3</sub>, and TFG extract amount. Physicochemical characterization of TFG-AgNPs was done on dynamic light scattering (DLS), field emission electron microscopy, energy dispersive X-ray spectroscopy, X-ray diffraction and Fourier transform infrared spectroscopy. The size determination studies using DLS revealed of TFG-AgNPs size between 95 and 110 nm. The antibacterial activity was studied against Escherichia coli, Proteus vulgaris, Pseudomonas aeruginosa and Staphylococcus aureus. The biosynthesized TFG-AgNPs showed remarkable anticancer efficacy against skin cancer cell line, A431 and also exhibited significant antioxidant efficacy. The conclusion of this paper in this study, we have demonstrated the utility of TFG seed extract as a reducing and capping agent that can efficiently reduce silver ions to AgNPs. The TFG AgNPs were thus characterised using UV-vis spectroscopy, SEM, DLS and so on. The AgNPs biosynthesised using 1 mM of AgNO3 reduced at 10:1 ratio of AgNO3: extract at 25°C for 15 min showed excellent antibacterial activity against Gram positive bacteria. Further, the TFG-AgNPs were observed to possess effective anticancer and antioxidant properties. This study, suggests that biosynthesis of

AgNPs using TFG seed extract could be a conventional and eco-friendly method in contrast to traditional chemical and physical synthesis [74].

## 2.16.17. Effect of difference concentrations of Al on the optical properties of AZO thin films

Effect of difference concentrations on the optical properties of AZO thin films, prepared on glass slides by Sol-gel method .The optical characteristics of the prepared thin films have been investigated by UV/Vis spectrophotometer in the wavelength range (200 - 800) nm .The films have a direct allow electronic transition with optical energy (Eg) values decreased from 3.29eV for AZO 1% thin films to 3.28 eV for the AZO 2% films and to 3.04eV for the AZO 3% films. The maximum value of the refractive index (n) for all thin films are given about 2.13. Also the extinction coefficient (K) and the real and imaginary dielectric constants  $(\in_1 \& \in_2)$ . The results indicate the films have good characteristics for optoelectronic applications. The conclusion of this paper ZnO thin films deposited by Sol-gel method shows band gap3.69eV which under AZO 1% concentration was found to be 3.29eV and 3.28eV at AZO 2% concentration. Under these treatments the film shows a shift of 0.41eV for AZO 2% concentration and 3.04eV for AZO 3% concentration in its optical spectra .Such dependence has been attributed to the structure of the film. The extinction coefficient value was increased in the UV region with treatment. The films give refractive index value equal to2.13 in the 345.39 (UV-region). Hence, these treatment for thin film give a best optical properties to be used for optoelectronic Applications [75].

#### 2.16.18. Methods of synthesis of nanoparticles and its applications

This work done by S. Kandasamy and \*R. Sorna Prema. In this study The aim of this review paper is to give an overview of the development and implications of nano particles synthesis which is an emerging field that covers a wide range of

applications. It play a major role in the development of innovative methods to produce new products to suitable existing production equipment and to reformulate new material and chemicals with improved performance resulting in less consumption of energy and material and reduce harm to the environment as well as environmental remediation. Nano catalyst is an expected to be fruitful areas which are synthesized recently by different methods. Current applications and research into the use of Nano catalyst in waste water management, textile, agriculture, and medicine has also been reviewed. The conclusion of this paper is Nano catalyst provides an extremely attractive platform in the Nano technology. In this review we express the Nano catalyst synthesis by various method and its applications in the food processing, packaging, medicine, antibacterial activity and waste water effluent treatment, however these applications are at in an elementary stage only. Research in Nanotechnology is estimated to have a huge impact on the development of new catalysts. The detailed understanding of Nanostructures and the ability to control size of materials will ensure a rational and cost efficient development of new and more capable catalyst [76].

## 2.16.19. Green Synthesis of Gold and Silver Nanoparticles Using (Averrhoa bilimbi Fruit Extract).

This work done by R. S. Rimal Isaac, In this study We report on rapid one-step green synthesis of gold and silver nanoparticles using fruit extract of *Averrhoa bilimbi Linn*. UV-Vis absorption spectroscopy was used to monitor the quantitative formation of gold and silver nano particles. The characteristics of the obtained gold and silver nanoparticles were studied using UV-Vis absorption spectroscopy (UV/Vis), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), and Energy-dispersive spectroscopy (EDX). UV/Vis spectrum showed Surface Plasmon Resonance (SPR) for both gold and silver nanoparticles at 540 and 420 nm. The EDX spectrum of the solution containing gold and silver

nanoparticles confirmed the presence of elemental gold and silver signals. The average diameter of the prepared nanoparticles in solution was about 50–150 nm. Synthesized particles were either hexagonal or rhomboidal in shape. This synthesis approach of gold and silver nano particles is cost effective and can be widely used in biological systems. The effect of fruit extract and metal ion concentration was also studied, the conclusion of this paper is Here, we have demonstrated a low-cost and one-pot green synthesis approach for preparation of stable gold and silver nano particles. Gold and silver nanoparticles were synthesized by a rapid, eco-friendly (biogenic, green route) process using Averrhoa bilimbi Linn. fruit extract as a reducing agent. The alcohols, amines, and phenols present in the fruit extract might have caused the reduction and capping of the nanoparticles. The characteristics of the obtained gold and silver nanoparticles were studied using UV-Vis absorption spectroscopy (UV/Vis), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM), and Energy dispersive spectroscopy (EDX). The EDX spectrum of the solution containing gold and silver nanoparticles confirmed the presence of elemental gold and silver signals along with aluminum (Al) as impurities coming from the sample substrate. The average diameter of the prepared nanoparticles in solution was about 50–150 nm. The present method eludes the use of toxic chemicals for the synthesis of gold and silver nanoparticles so it can be used for biological applications. Synthesized particles were either hexagonal or rhomboidal in shape. This synthesis approach of gold and silver nanoparticles is cost effective and can be widely used in biological systems. Nanoparticles of free metals have been extensively researched because of their unique physical properties, chemical reactivity and potential applications in catalysis, biological labeling, biosensing, drug delivery, antibacterial and antiviral activity, detection of genetic disorders, gene therapy, and DNA sequencing [77].

#### 2.16.20. Green synthesis of ZnO nanoparticles by Calotropis Gigantea

This work done by Vidya Ca, Shilpa Hirematha\*, In this study Present study focuses on the green synthesis of ZnO nanoparticles by zinc nitrate and utilizing the bio components of leaves extract of Calotropis Gigantea. The ZnO nano crystallites of average size range of 30-35 nm have been synthesized by rapid, simple and ecofriendly method. Zinc nanoparticles were characterized using scanning electron microscopy (SEM) and X-ray diffraction (XRD). The particles obtained are spherical in nature and are agglomerates of nanocrystallite. The X ray patterns show hexagonal crystal type for ZnO. The results coincide with literature XRD pattern for hexagonal wurtzite ZnO. The size of nano crystallites is calculated by considering XRD data by Debye-Scherrer's Formula, The conclusion of this paper is The rapid biological synthesis of zinc nanoparticles using leaf extract of Calotropis gigantea provides an environmental friendly, simple and efficient route for synthesis of nanoparticles. The use of plant extracts avoids the usage of harmful and toxic reducing and stabilizing agents. The synthesized nano crystallites of ZnO are in the range of 30-35 nm. Zinc nanoparticles can exist in ions only in the presence of strong oxidizing substances. The environmental conditions will affect the stability of nano particle and agglomerates are formed. The synthesis of ZnO nano particles is still in its infancy and more research needs to be focused on the mechanism of nanoparticle formation which may lead to fine tuning of the process ultimately leading to the synthesis of nanoparticles with a strict control over the size and shape parameters. [78]

## **CHAPTER THREE**

### **MATERIAL AND METHOD**

#### 3.1 Introduction

This chapter speaks about of all material used for preparation of samples of neem trigonella foenum graecum, negella sativa and elettaria cardamomum seeds were powdered and dissolved in distilled water, ethanol and methanol respectively. Determinations of their optical properties, structure were done using UVspectrometer, FTIR and XRD. The powder of these seeds were added to water then it's PH and the Living E-coli were found.

#### **3.2.** Materials:

## 3.2.1. Azadirchta Indica seeds (Neem seeds)

Neem is an attractive broad-leaved, evergreen tree which can grow up to 30m tall and 2.5m in girth. Its trunk usually straight is 30-80 cm in diameter. Its spreading branches form a rounded crown of deep-green leaves and honey-scented flowers as much as 20m across. Neem - the legendary medicinal tree of india has grown with the human settlement all over the country and has been an integral part of the human settlement all over the country and has been an integral part of the Indian way of life for centuries. The history of the Neem tree is inextricably linked to the history of the Indian civilization. The Neem tree has for a very long time been a friend and protector of the Indian villager. For ages Indians have trusted this tree to fortify their health and remedy scores of diseases. In addition, it has been used for protecting food and stored grains and as a fertilizer and natural pesticide for the fields. It has been used for a far wider array of uses than any other tree! Neem seeds are also described as anthelminitic, antileprotic, antipoisonous and bitter in taste. Seed is very important both because of its high lipid content as well

as the occurrence of a large number of bitter principles (azadirachtin, azadiradione, fraxinellone, nimbin, salannin, salannol, vepinin, vilasinin, etc.) in considerable quantities. Azadirachtin has proven effectiveness as a pesticide against about 200 insect species and is reported as non-toxic to humans. Neem kernel lipids are similar to the normal glycerides from other oilseeds and contains oleic acid (50-60%), palmitic acid (13-15%), stearic acid (14-19%), linoleic acid (8- 16%) and arachidic acid (1-3%). It is brownish yellow, non-drying oil with an acrid taste and unpleasant odour. The quality of the oil differs with the method of processing.



Figure (3.1): Dry seed of azadirchta indica seeds (Neem seeds)

## 3.2.2. Trigonella Foenum Graecum seeds

Fenugreek (Trigonella Foenum Graecum) is an annual herb that belongs to the family leguminosae widely grown in Pakistan, India, Egypt, and Middle Eastern countries by Alarcon-Aguilara et al. Fenugreek in one such plant whose leaves and seeds are widely consumed in Pakistan, India and other oriental countries as a spice in food preparations due to their strong flavor and aroma, and as an ingredient in traditional medicine. It is rich source of calcium, iron,  $\beta$ carotene and other vitamins by Sharma et al. Both leaves and seeds should be included in normal diet of family, especially diet of growing kids, pregnant ladies, puberty reaching girls and elder members of family because they have haematinic (i.e. blood formation) value by day. Fenugreek seed is widely used as a galactagogue (milk producing agent) by nursing mothers to increase inadequate breast milk supply by Fleiss. The seeds of fenugreek contain lysine and Ltryptophan rich proteins, mucilaginous fiber and other rare chemical constituents such as saponins, coumarin, fenugreekine, nicotinic acid, sapogenins, physics acid, scopoletin and trigonelline, which are thought to account for many of its presumed therapeutic effects may inhibit cholesterol absorption and thought to help lower sugar levers by Billaud, Sauvaire et al and Sauvaire. Therefore, fenugreek seeds are used as a traditional remedy for the treatment of diabetes and hypercholesterolemia in Indian and Chinese medicine by Basch et al and Miraldi et al. It's reported to have restorative and nutritive properties and to stimulate digestive processes, useful in healing of different ulcers in digestive tract by Khoslaetal [8].



Figure (3.2): Dry seed of Trigonella Foenum Graecum.

## 3.2.3 Negella sativa seeds

Negella Sativa (NS) or black cumin (BC) is a globally cultivated plant which is majorly found in southwest Asia, southern Europe, and northern Africa. The seeds of BC are well known for their medicinal uses and exhibit strong antimicrobial activities particularly for gram-positive bacteria like Staphylococcus aureus and Vibrio cholera BC seeds (BCS) contain traces of alkaloids, number of esters of structurally unusual unsaturated fatty acids with terpene alcohols Based on dry weight, the chemical composition of BC was observed as: fiber 21.98%, lipid 28.91%, carbohydrate 15.57%, ash 4.00%, protein 22.00%, magnesium 0.25%, calcium 0.36% and phosphorus 0.72% while moisture was 5.54%. The total energy was observed to be 4.18 k.cal g<sup>-1</sup>. The presences of the above organic compounds in BCS provide different functional groups to seeds surface and it can be suggested that seeds may eventually provide attractive sites for charged molecules and/ or ions. BCS were utilized as an affordable and sustainable material for the bio-adsorptive removal of several water pollutants.



Figure (3.3): Dry seed of negell sativa

## 3.2.4. Elettaria Cardamomum seeds

also known as 'Elaichi', is an important member of household spices in India, best known for its aromatic and medicinal properties. Elettaria seed berry has numerous applications in everyday food items and are quite expensive and precious.



Figure (3.4): Dry seed of Elettaria Cardamomum

#### 3.2.5 Methanol

Methanol, also known as methyl alcohol amongst other names, is a chemical and the simplest alcohol, with the formula CH<sub>3</sub>OH (a methyl group linked to a hydroxyl group, often abbreviation (MeOH). It is a light, volatile colorless, flammable liquid with distinctive alcoholic odors similar to that of ethanol (potable alcohol).

#### **3.2.6.** Ethanol

Ethanol is a natural byproduct of plant fermentation and also can be produced through the hydration of ethylene. Ethanol, also called alcohol with chemical formula  $C_2H_6O$ , ethyl alcohol and grain alcohol, is a clear, colorless liquid and the principle ingredient in alcoholic beverages like beer, wine or brandy. Because it can readily dissolve in water and other organic compounds, ethanol also in an ingredient in a range of products, form personal care and beauty products to paints and varnishes to fuel.

## **3.2.7. Distilled water**

Distilled water is water that has been boiled into vapor and condensed back into liquid in a separate container. Impurities in the original water that do not boil bellow or near the boiling point of water remain in the original container. Thus distilled water is a type of purified water ( $H_2$  O).

## **3.2.8. PH meter**

To determine the PH of the synthesized nanoparticles and the reaction mixtures, systronic digital PH meter. Model MAC (MSW-552) was used with maximum uncertainty in PH of  $\pm 0.01$  unit. To determine the PH of solution first calibrate the pH meter using buffer solutions of different PH such as 7.0, 4.0, and 9.2. Thoroughly wash the PH electrode between measurements with distilled water to avoid carryover impurity of the tested solutions. Softly blot the electrode on a tissue paper to remove the excess rinse water and do not rub the bulb to avoid build-up static charge [9].



Figure (3.5): shows the pH meter.

### **3.3.** Characterization Techniques

The material Characterization Lap has a wide variety of tools in terms of their structural, composition and optical properties. The crystal structure of the samples was characterized at room temperature by using a Philips PW1700 X-ray Diffraction. The location of band position of the elements were examined using the Fourier Transform Infrared spectrophotometer (FTIR) in the rang 400 to 4000 cm<sup>-1</sup> to record some location of the band positions. The optical properties were examined by using UV-visible spectroscopy.

#### **3.3.1. X-ray Diffraction for structural Analysis**

X-ray is an electromagnetic radiation of wavelength about  $1A^0 (10^{-10} \text{ m})$ , which is about the same size as an atom. They occur in that portion of the electromagnetic spectrum between gamma –rays and the ultraviolet. X-rays provides valuable tool for the Lattice structure of a crystalline substance like unit cell dimensions, bond angles. The x-ray diffraction (XRD) device utilizes the principle of diffraction to obtain crystal spacing between successive planes and the nano crystal size as well.

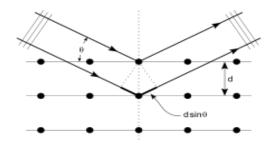


Figure (3. 6): illustrates the reflection of X-rays from two planes of atoms in solid.

A crystal Lattice is a regular three- dimensional distribution (cubic, rhombic, etc) of atoms in space. These are arranged so that they form a series of parallel planes separated from one another by a distance (d), which varies according to the nature of the material. For any crystal, planes exist in a number of different orientations, each with its own specific d-spacing. When a monochromatic X-ray beam with wavelength ( $\lambda$ ) is projected onto a crystalline material at an angle theta, diffraction occurs only when the distance traveled by the rays reflected from successive planes differs by a complete number (n) of wavelengths, which leads to famous Bragg's law:

$$n\lambda = 2dsin\theta \tag{3.1}$$

Where (n) is an integer 1, 2, 3... (Diffraction order), ( $\lambda$ ) the wavelength of the incident X-ray, (d) is the interatomic spacing in angstroms,, and  $\theta$  is the Bragg angle. By varying the angle theta, the Bragg's Law conditions are satisfied by different d-spacing in polycrystalline materials. Plotting the angular positions and intensities of the resultant diffract-gram is formed by addition of the individual patterns. Based on the principle of X-ray diffraction, a wealth of structural information about the material investigated can be obtained. By altering the various parameters like the geometry of incident rays and the orientation of the detector and crystal, one can obtain all possible diffraction directions of the Lattice [17, 18]. X-ray diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined. X-ray diffraction (XRD) is a non-destructive technique for analyzed the structure of materials, primarily at the atomic or molecular level. It works best for materials that are crystalline or partially crystalline (i.e., that have periodic structure order) but is also used to study noncrystalline nano materials. XRD relies on the fact that X-rays have wavelengths on the order of nano meters.



Figure (3.7): X-Ray Diffract meter: XRD (Wavelength 1.54A<sup>0</sup>)

# **3.3.2.** Fourier Transform Infrared spectrometer (FTIR) for optical properties

The physics of individual atoms and simple molecules can be understood by studying their interaction with electromagnetic radiations. This study is called as spectroscopy. In spectroscopy, electromagnetic radiation of a particular frequency or a range of frequencies is allowed to fall on a sample and then analyzed in terms of the intensity at different frequencies. This indicates about the line absorbed or emitted by the molecule, and hence giving a picture of the molecular energy levels. In the region of longer wavelength or low frequency the identification of different types of chemicals by possible use the technique of infrared spectroscopy and the instrument requires for its execution is Fourier Transform Infrared (FTIR) spectrotometer as shown figure (3.8). This spectroscopy is merely based on the fact that molecules absorb specific frequencies that are characteristic of their structure termed as resonant frequencies, i.e. the frequency of the absorbed radiation matches the frequency of the bond or group that vibrates. As each different material is a unique combination of atoms, no two compounds produce the exact same infrared spectrum. Therefore, infrared spectroscopy can result in a positive identification (qualitative analysis) of every different kind of material. Present [19,20]. FTIR can be used to analyze a wide variety of materials in bulk or thin films, liquids, solids, pastes, powders, and other forms. FTIR analysis can be used to analyze samples up to  $\sim 11$  millimeter in diameter, and either measure in bulk or the top  $\sim$  1 micrometer layer.



Figure (3.8): shows the Fourier Transform Infrared spectrometer.

#### 3.3.3. UV- Visible Spectrometer for optical properties

Ultraviolet and visible (UV/V) spectrometers have become the most important analytical instrument in the modern day laboratory. In many applications other techniques could be employed but none rival UV/V spectrometry for its simplicity, versatility, speed, accuracy and effectiveness. UV/V spectrometer is a tool used for the investigation of electronic transitions and hence to determine the electronic bonding in a molecule. In Ultraviolet / visible (UV/V) spectrometer, electromagnetic radiation is emitted within the wavelength range of about 200-800 nm from the source. The source is often a Deuterium (or hydrogen) lamp, a tungsten filament lamp, or a xenon arc lamp. The radiation from the source is then passed through a wavelength selector (either a diffraction grating or filter) through which a narrow band of wavelength can pass. In a standard UV/V spectrometer, the beam of light is split into two parts by a partial mirror, one half of the beam (the sample beam) is directed through a transparent cell containing a solution of the compound being analyzed, and one half (the reference beam) is directed through an identical cell that does not contain the compound but contains the same solvent wherein the compound is dissolved. The beam from the wavelength selector passes through the sample and is absorbed by the sample according to Beer's Law. The difference of light intensities scanned over a range of wavelength are taken and

compared. The intensity profiles over a range of wavelength are observed. In complex molecules the energy levels are more closely spaced and photons of near ultraviolet and visible light can affect the transition. These substances, therefore, will absorb light in some areas of the near ultraviolet and visible regions.



Figure (3.9): shows the Ultraviolet / visible (UV- V) spectroscopy

#### 3.4. Method

#### **3.4.1. Preparation of Samples:**

Trigonella foenum graecum seeds were collected from the local market. The T. foenum graecum (seed) was powdered finely using morter and pestle. Five gram powder was dissolved in 250 mL beaker Including 25 mL (Distilled water, ethanol and methanol) for 72 hours. The neem seeds were collected from the local abofroa city. The neem (seed) was powdered finely using morter and pestle, 5gram powder was dissolved in 250 mL beaker including 25 mL (distillation water, ethanol and methanol) for 72 hours. The negilla sativa seeds were collected from the local market. The negella Sativa (seed) was powdered finely using morter and pestle.

Then five gram powder was dissolved in 250 mL beaker Including 25 mL (distilled water, ethanol and methanol) for 72 hours. The elettaria cardamomum seeds were collected from the local market. The elettaria cardamomum (seed) was powdered finely using morter and pestle. Then five gram powder was dissolved in 250mL beaker including 25mL (Distilled water, ethanol and methanol) for 72 hours. The prepared extracts were studied by using UV/V, FTIR and XRD techniques.

#### **3.4.2. Experimental procedures**

- 1. The four seeds (Trigonella Foenum Graecum (neem), trigonella foenum graecum, negella sativa, and elettaria cardamomum) were grinded to be in a powder form.
- 2. An equal amount from the powder of each seed was dissolved in three solvent. These solvents are water, methanol and ethanol. Thus one have 12 samples.
- 3. The UV/V spectrometer was used to display the absorption spectra were used to display spectra for reflection, transmittance, refractive index, and extension coefficients beside optical and electrical conductivity for all samples. These figures were used to find the electric permittivity and magnetic permeability for all samples.
- 4. The FTIR spectrum for the four seeds powders were displayed using FTIR spectrometer.
- 5. An Equal amount from the extracts from the 12 samples described in step two was added to water. Then the water PH and the living E-coli counts were found for the 12 samples.



Figure (3.10): shows the solution of the samples.

### **Chapter Four**

### **RESULTS AND DISCUSSION**

#### **4.1. Introduction**

This chapter displays the results after chactraized samples using XRD, UV-VIS spectroscopy and FTIR spectroscopy.

#### 4.2. X-ray diffraction (XRD) Results of all samples

The powders of the four seeds were examined by XRD device to determine their crystal spacing and nano size beside their crystal form as shown in the figures and table below.

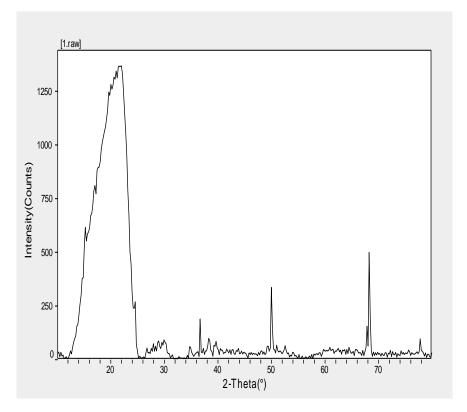


Figure (4.1): XRD spectrum of Azadirchta Indica be for sample.

Table (4.1) Lattice Constants from Peak Locations and Miller Indices [monoclinicprimitive] of Azadirachta Indica (neem).

|          |     | 20      |      | Ċ      | l (10 <sup>-10</sup> m) | h              | k   | 1               | X <sub>s</sub> (nm)   |
|----------|-----|---------|------|--------|-------------------------|----------------|-----|-----------------|-----------------------|
|          |     | 20.063  |      | 4.4221 |                         | 1              | 0   | 2               | 100                   |
| a= 9.976 | b : | = 7.294 | c= 6 | .291   | *α=90                   | <sup>o</sup> β | = 1 | 07 <sup>0</sup> | $^{O}\gamma = 90^{O}$ |

Density =  $2.2168 \text{ mg.cm}^{-3}$ 

Crystal Form: Monoclinic –Primitive.

Space Group: P21/n (14) (Y-unique).

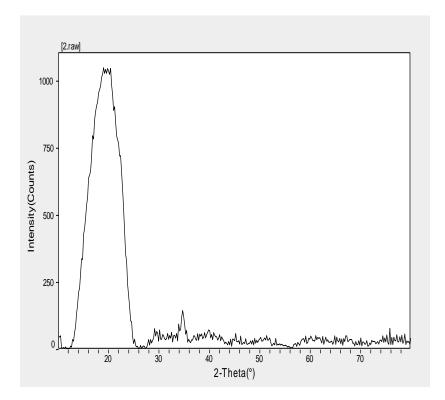


Figure (4.2): XRD spectrum of fenugreek (trigonella) after sample.

Table (4.2): Lattice constants from peak locations and miller indices [Tetragonal – Primitive] of fenugreek.

|           |     | 20             | d ( 10 <sup>-10</sup> m ) | h k l                      | X <sub>s</sub> (nm) |
|-----------|-----|----------------|---------------------------|----------------------------|---------------------|
|           |     | 19.871         | 3.7831                    | 02 0                       | 27.3                |
| a = 7.574 | b = | = 7.754 c= 10. | 216 α                     | $\beta = \beta = \gamma =$ | 90 <sup>0</sup>     |

Density =0.8901 mg.cm<sup>-3</sup>

Crystal Form: Tetragonal – Primitive.

Space Group: unknown.

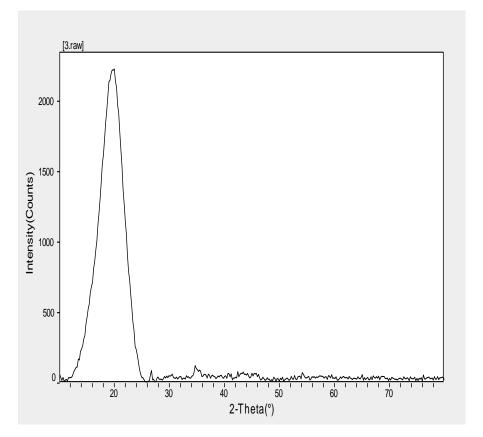


Figure (4. 3): XRD spectrum of nigella sativa before sample.

Table (4.3): Lattice constants from peak Locations and miller indices [Triclinic – Primitive] of nigella sativa .

| 20     | d ( 10 <sup>-10</sup> m ) | h k l | X <sub>s</sub> (nm) |
|--------|---------------------------|-------|---------------------|
| 19.191 | 4.621                     | 1 2 7 | 11.1                |

a=7.01 b=7.95 c= 7.63

 $\alpha = 100^{0}\beta = 120^{0}\gamma = 80^{0}$ 

Density =  $2.3341 \text{ mg. cm}^{-3}$ 

Crystal form: Triclinic –Primitive.

Space Group: p - 1(2).

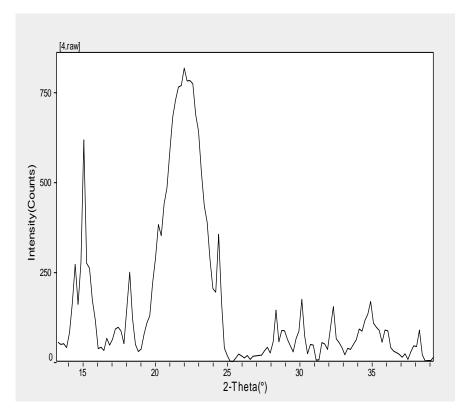


Figure (4.4): XRD spectrum of elettaric cardomum after sample.

Table (4.4): Lattice constants from peak locations and miller indices [cubic – Primitive] of elettaric cardomum .

| 20     | d ( 10 <sup>-10</sup> m ) | h k l | X <sub>s</sub> (nm) |
|--------|---------------------------|-------|---------------------|
| 22.367 | 3.9715                    | 00 2  | 48.4                |

a = b = c = 10.31

 $\alpha = \beta = \gamma = 90^{\rm O}$ 

Density =  $2.0225 \text{ mg.cm}^{-3}$ .

Crystal Form: cubic –Primitive.

Space Group: unknown.

#### 4.3. Fourier Transforms Infrared Spectroscopy (FTIR Results of all samples.

The FTIR spectrometer was used to determine the chemical bonds for the four seeds powder as shown in the figures and table below.

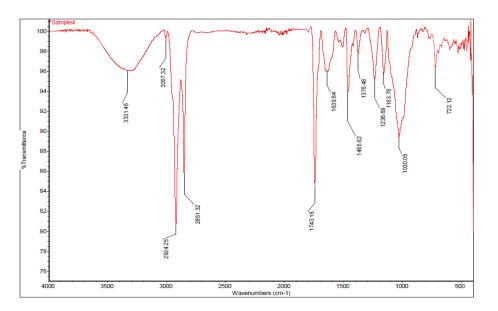


Figure (4.5): Fourier transform Infrared spectroscopy (FTIR) spectrum of Azadirchta Indica seeds.

| Wave number(frequency) cm <sup>-1</sup> | Bond (Remarks)        | functional group                           |
|---|-----------------------|--|
| 722.12                                  | С–Н "оор"             | Aromatics                                  |
| 1030.05                                 | C–N stretch           | aliphatic amines                           |
| 1163.76                                 | C–O stretch           | alcohols, carboxylic acids, esters, ethers |
| 1236.69                                 | C–O stretch           | alcohols, carboxylic acids, esters, ethers |
| 1376.48                                 | O-H bending           | Phenol                                     |
| 1465.62                                 | C–H bend              | Alkanes                                    |
| 1639.84                                 | N–H bend              | 1° amines                                  |
| 1743.16                                 | C=O stretch           | esters, saturated aliphatic                |
| 2851.32                                 | C–H stretch           | Alkanes                                    |
| 2924.25                                 | C–H stretch           | Alkanes                                    |
| 3007.32                                 | =C–H stretch          | Alkenes                                    |
| 3331.46                                 | O-H stretch, H-bonded | alcohols, phenols                          |

Table (4.5): Characteristic IR Absorptions of Azadirchta Indica seeds:

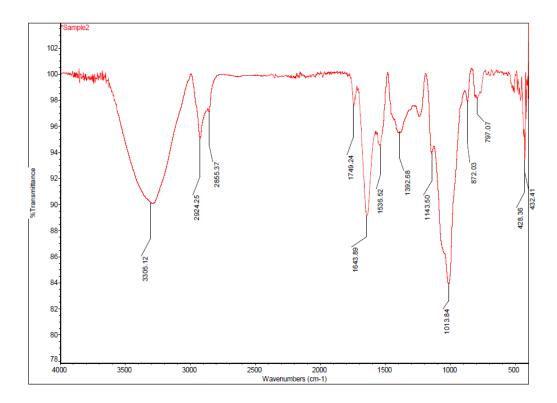


Figure (4.6): Fourier transform Infrared spectroscopy (FTIR) spectrum of Trigonella Foenum Graecum seeds.

| <i>Wave number( frequency) cm</i> <sup>-1</sup> | Bond (Remarks)                 | functional group            |  |
|---|--------------------------------|-----------------------------|--|
| 432.41  | Fe <sub>2</sub> O <sub>3</sub> | Hematite                    |  |
| 428.36  | Fe <sub>2</sub> O <sub>4</sub> | Magnetite                   |  |
| 797.07  | С–Н "оор"                      | Aromatics                   |  |
| 872.03  | С–Н "оор"                      | Aromatics                   |  |
| 1013.84   | C–N stretch                    | Alkyl halides (R-F)         |  |
| 1143.50   | C–N stretch                    | aliphatic amines            |  |
| 1392.68   | O-H bending                    | Alcohol                     |  |
| 1536.52   | N–O asymmetric stretch         | nitro compounds             |  |
| 1643.89   | N–H bend                       | 1° amines                   |  |
| 1749.24   | C=O stretch                    | esters, saturated aliphatic |  |
| 2855.37   | C–H stretch                    | Alkanes                     |  |
| 2924.25   | C–H stretch                    | Alkanes                     |  |
| 3305.12   | –C≡C–H: C–H stretch            | alkynes (terminal)          |  |

Table (4.6): Characteristic IR Absorptions of Trigonella Foenum Graecum seeds.

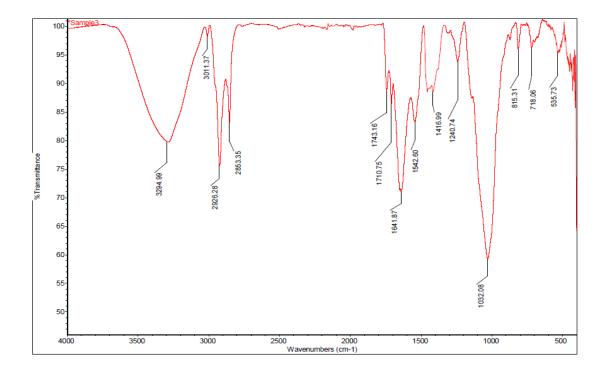


Figure (4.7): Fourier transform Infrared spectroscopy (FTIR) spectrum of negella sativa seeds.

| Wave number( frequency) cm <sup>-1</sup> | Bond (Remarks)         | functional group                               |
|--|------------------------|--|
| 535.73                                   | C–Br stretch           | alkyl halides                                  |
| 718.06                                   | С–Н "оор"              | Aromatics                                      |
| 815.31                                   | С–Н "оор"              | Aromatics                                      |
| 1032.08                                  | C–N stretch            | aliphatic amines                               |
| 1240.74                                  | C–N stretch            | aliphatic amines                               |
| 1416.99                                  | C–C stretch (in–ring)  | Aromatics                                      |
| 1542.60                                  | N–O asymmetric stretch | nitro compounds                                |
| 1641.87                                  | N–H bend               | 1° amines                                      |
| 1710.75                                  | C=O stretch            | $\alpha,\beta$ -unsaturated aldehydes, ketones |
| 1743.16                                  | C=O stretch            | esters, saturated aliphatic                    |
| 2853.35                                  | C–H stretch            | Alkanes  |
| 2926.28                                  | C–H stretch            | Alkanes  |
| 3011.37                                  | =C–H stretch           | alkenes  |
| 3294.99                                  | C–H stretch            | Aromatics                                      |

Table (4.7): Characteristic IR Absorptions of negella sativa seeds.

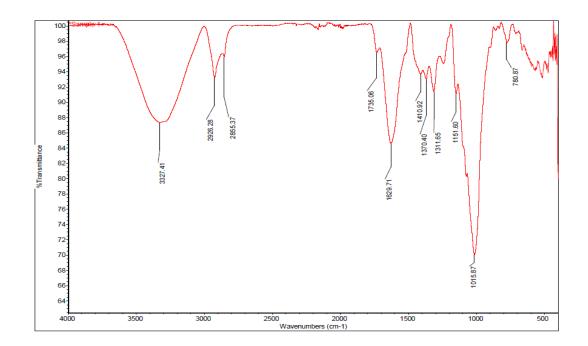


Figure (4.8): FTIR spectrum of elettaria cardamomum seeds.

| Wave number( frequency) cm <sup>-1</sup> | Bond (Remarks)        | functional group                           |
|--|-----------------------|--|
| 780.87                                   | С–Н "оор"             | Aromatics                                  |
| 1015.87                                  | C-F stretch           | Alkyl halides (R-F)                        |
| 1151.60                                  | C–N stretch           | aliphatic amines                           |
| 1311.65                                  | C–O stretch           | alcohols, carboxylic acids, esters, ethers |
| 1370.40                                  | C–H rock              | Alkanes                                    |
| 1410.92                                  | C–C stretch (in–ring) | Aromatics                                  |
| 1629.71                                  | N–H bend              | 1° amines                                  |
| 1735.06                                  | C=O stretch           | Aldehydes, saturated aliphatic             |
| 2855.37                                  | C–H stretch           | Alkanes                                    |
| 2926.26                                  | C–H stretch           | Alkanes                                    |
| 3327.41                                  | –C≡C–H: C–H stretch   | alkynes (terminal)                         |

Table (4.8): Characteristic IR Absorptions of Elettaria cardamomum seeds .

## 4.4. The UV spectra for the 12 samples formed from dissolving the four seeds in three solvents

### **4.4.1.** The UV spectra of Azadirchta Indica dissolved in (water, methanol and ethanol)

The UV spectra of the 12 samples for absorption, reflection, extension, refractive index optical and electrical conductivities were shown in the below figures.

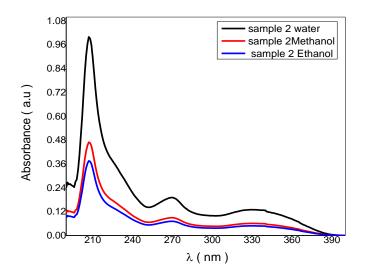


Figure (4.9): optical Absorbance spectra of the three samples of Azadirchta Indica dissolved by (water, methanol and ethanol)

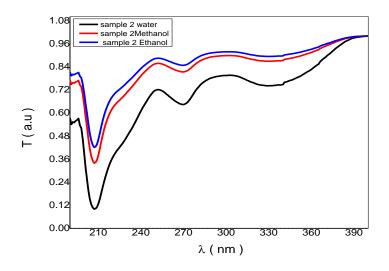


Figure (4.10): optical transmittance (T) Spectra of the three samples of Azadirchta Indica dissolved by (water, methanol and ethanol)

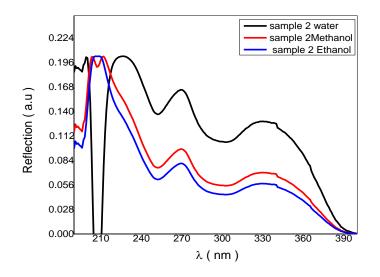


Figure (4.11): optical Reflection spectra of the three samples of Azadirchta Indica dissolved by (water, methanol and ethanol)

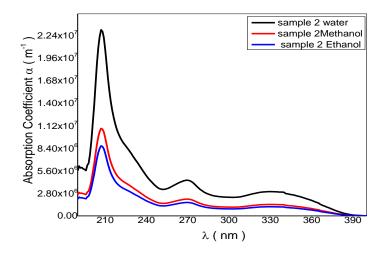


Figure (4.12) Optical Absorption coefficient spectra of spectra of the three samples of Azadirchta Indica dissolved by (water, methanol and ethanol)

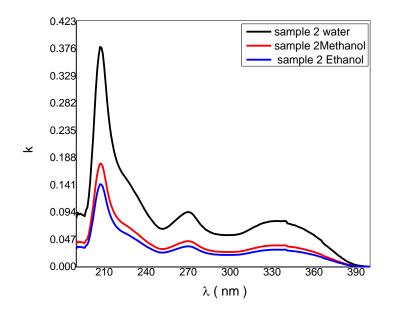


Figure (4.13): The variation of Extinction coefficient (K) with wavelength ( $\lambda$ ) for the three samples of Azadirchta Indica dissolved by (water, methanol and ethanol).

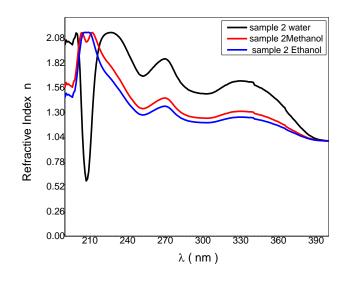


Figure (4.14): The Variation of refraction index (n) with wavelength of the three samples of Azadirchta Indica dissolved by (water, methanol and ethanol).

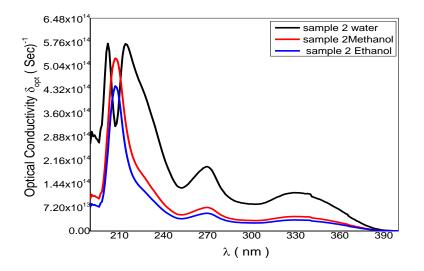


Figure (4.15): Plot of optical conductivity as a function of wavelength for the three samples of Azadirchta Indica dissolved by (water, methanol and ethanol).

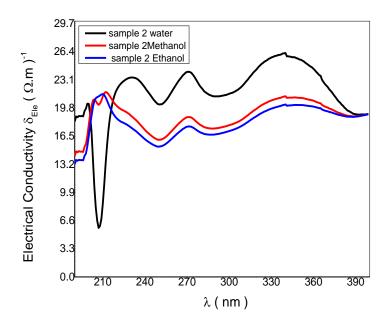


Figure (4.16): Plot of Electrical conductivity as a function of wavelength for the three samples of Azadirchta Indica dissolved by (water, methanol and ethanol).

**4.4.2.** The UV spectra of Trigonella Foenum Graecum dissolved in (water, methanol and ethanol) samples

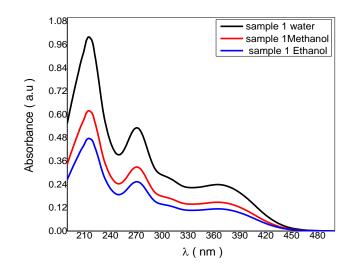


Figure (4.17): optical Absorbance spectra of the three samples of Trigonella Foenum Graecum dissolved by (water, methanol and ethanol).

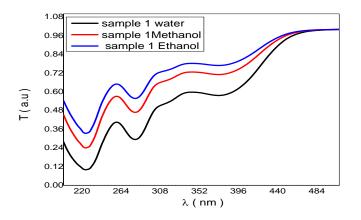


Figure (4.18): optical transmittance (T) Spectra of the three samples of Trigonella Foenum Graecum dissolved by (water, methanol and ethanol).

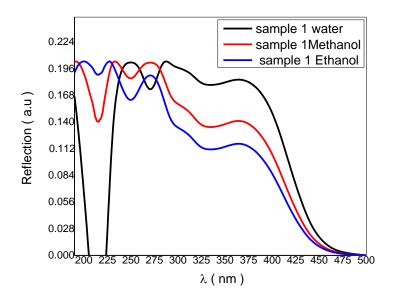
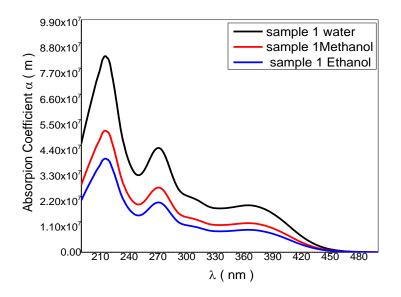
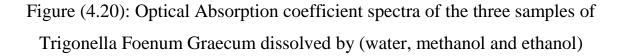


Figure (4.19): optical Reflection spectra of the three samples of Trigonella Foenum Graecum dissolved by (water, methanol and ethanol).





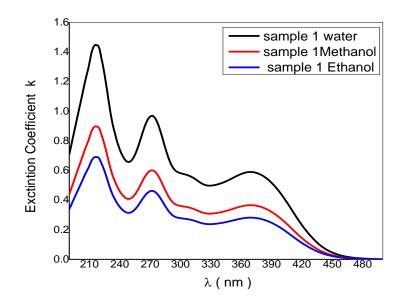


Figure (4.21): The variation of extinction coefficient (K) with wavelength ( $\lambda$ ) for the three samples of Trigonella Foenum Graecum dissolved by (water, methanol and ethanol).

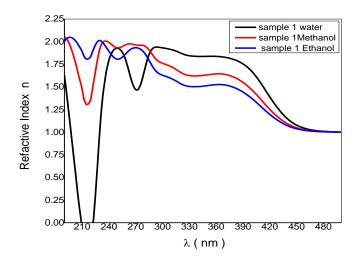


Figure (4.22): The Variation of refraction index (n) with wavelength of the three samples of Trigonella Foenum Graecum dissolved by (water, methanol and ethanol)

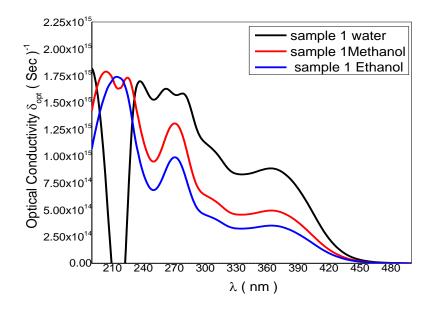


Figure (4.23): Plot of optical conductivity as a function of wavelength for the three samples of Trigonella Foenum Graecum dissolved by (water, methanol and ethanol)

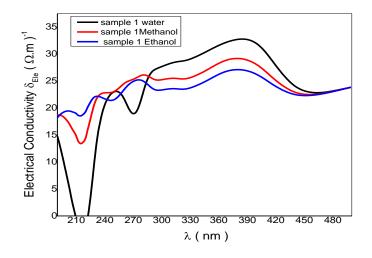
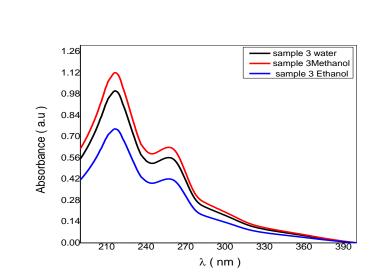


Figure (4.24): Plot of Electrical conductivity as a function of wavelength for the three samples of Trigonella Foenum Graecum dissolved by (water, methanol and ethanol)

**4.4.3.** The UV spectra of Negella sativa dissolved in (water, methanol and ethanol) samples



 $\setminus$ 

Figure (4.25): optical Absorbance spectra of the three samples of Negella sativa dissolved by (water, methanol and ethanol)

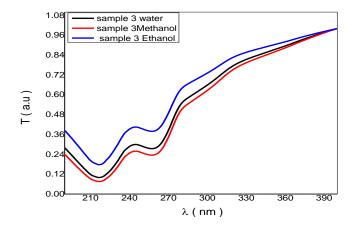


Figure (4.26): optical transmittance (T) Spectra of the three samples of Negella sativa dissolved by (water, methanol and ethanol).

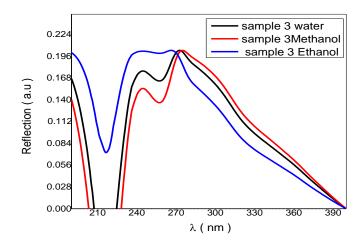


Figure (4.27): optical Reflection spectra of the three samples of Negella sativa by dissolved (water, methanol and ethanol)

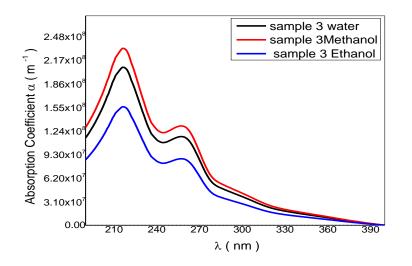


Figure (4.28): Optical Absorption coefficient spectra of spectra of the three samples of Negella sativa dissolved by (water, methanol and ethanol)

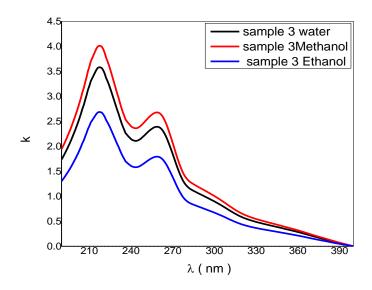


Figure (4.29): The variation of Extinction coefficient (K) with wavelength ( $\lambda$ ) for the three samples of Negella sativa dissolved by (water, methanol and ethanol).

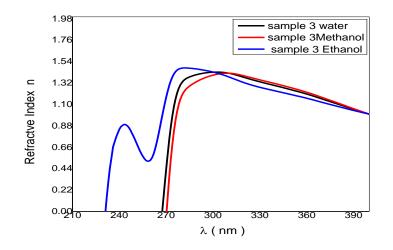


Figure (4.30): The Variation of refraction index (n) with wavelength of the three samples of Negella sativa dissolved by (water, methanol and ethanol).

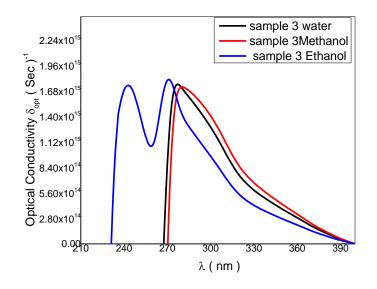


Figure (4.31): Plot of optical conductivity as a function of wavelength for the three samples of Negella sativa dissolved by (water, methanol and ethanol)

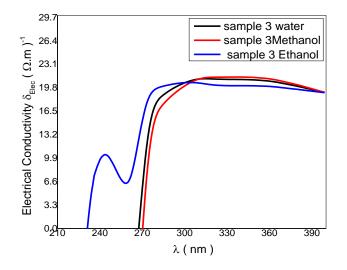


Figure (4.32): Plot of Electrical conductivity as a function of wavelength for the three samples of negella sativa dissolved by (water, methanol and ethanol).

## **4.4.4.** The UV spectra of Elettaria cardamomum dissolved in (water, methanol and ethanol) samples

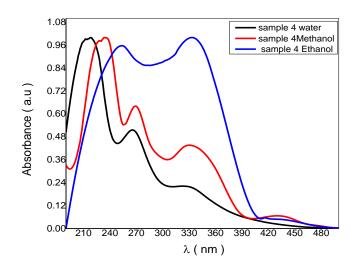


Figure (4.33): optical Absorbance spectra of the three samples of Elettaria cardamom dissolved by (water, methanol and ethanol)

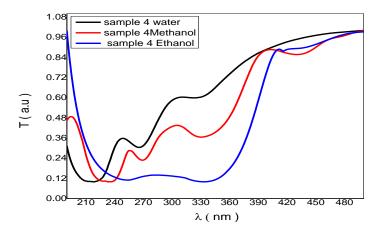


Figure (4.34): optical transmittance (T) Spectra of the three samples of Elettaria cardamomum dissolved by (water, methanol and ethanol).

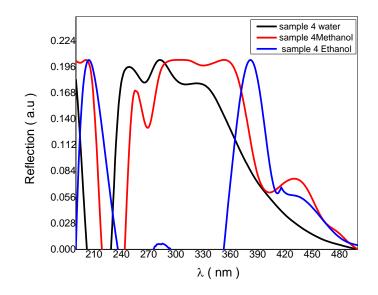


Figure (4.35): optical Reflection spectra of the three samples of Elettaria cardamomum dissolved by (water, methanol and ethanol).

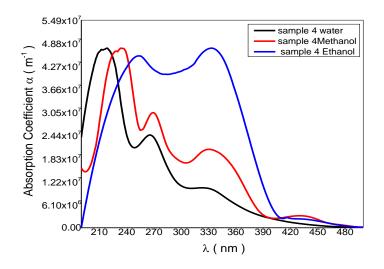


Figure (4. 36): Optical Absorption coefficient spectra of spectra of Elettaria cardamomum dissolved by (water, methanol and ethanol).

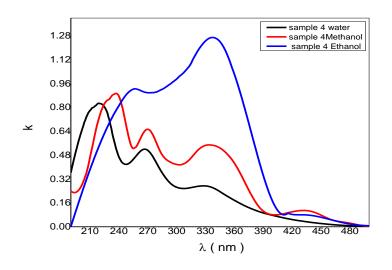


Figure (4.37): The variation of Extinction coefficient (K) with wavelength ( $\lambda$ ) for the three samples of Elettaria cardamomum dissolved by (water, methanol and ethanol)

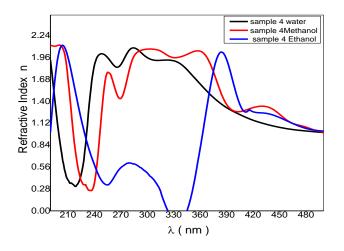


Figure (4.38): The Variation of refraction index (n) with wavelength of the three samples of Elettaria cardamomum dissolved by (water, methanol and ethanol)

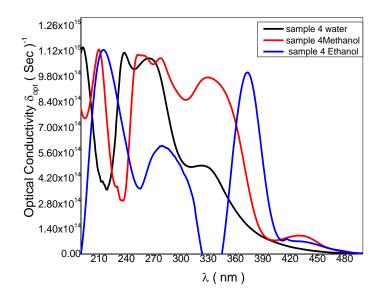


Figure (4.39): Plot of optical conductivity as a function of wavelength for the three samples of Elettaria cardamomum dissolved by (water, methanol and ethanol).

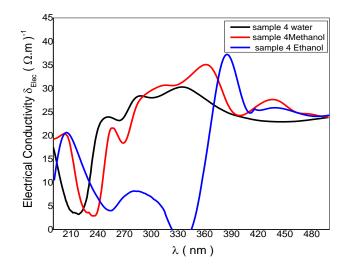


Figure (4.40): Plot of Electrical conductivity as a function of wavelength for the three samples of Elettaria cardamomum dissolved by (water, methanol and ethanol).

#### 4.5. The PH and E-coli Bacteria counts for the 12 samples of water

When the four seeds powders were dissolved in the three solvents they form 12 samples. An equal amounts from each sample were added to water to form 12 water samples. The PH and the E-coli count existing already in water was found as shown by the figures and tables below.

| Sample    | Water mixture       | First Week | Second | Third Week | Fourth week |
|-----------|---------------------|------------|--------|------------|-------------|
|           |                     |            | Week   |            |             |
| Sample C  | Control             | 14.75      | 12.21  | 11.83      | 9.95        |
| sample 1  | Neem ethanol        | 14.64      | 11.63  | 10.24      | 8.95        |
| sample 2  | Neem methanol       | 14.52      | 10.411 | 9.12       | 8.83        |
| sample 3  | Neem water          | 14.48      | 10.21  | 8.05       | 7.73        |
| sample 4  | T.F.G ethanol       | 14.29      | 9.93   | 7.81       | 7.52        |
| sample 5  | T.F.G methanol      | 14.13      | 8.72   | 7.69       | 6.46        |
| sample6   | T.F.G water         | 14.05      | 8.54   | 7.34       | 6.23        |
| sample 7  | N.Sativa ethanol    | 14.02      | 7.36   | 6.24       | 5.95        |
| sample 8  | N.Sativa methanol   | 14.53      | 11.42  | 10.13      | 8.73        |
| sample 9  | N.Sativa water      | 14.36      | 10.09  | 8.02       | 7.58        |
| sample 10 | E.cardamom ethanol  | 14.09      | 8.58   | 7.75       | 6.34        |
| sample 11 | E.cardamom methanol | 14.01      | 8.43   | 7.23       | 6.11        |
| sample 12 | E.cardamom water    | 14         | 8.33   | 7.20       | 6.09        |

Table (4.9): The PH of the 12 water sample beside control.

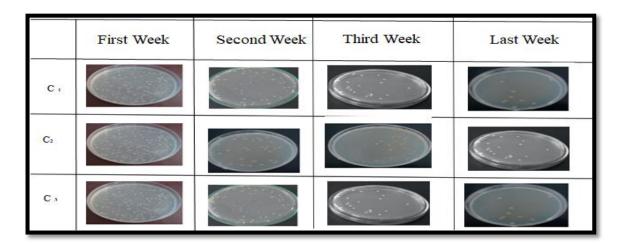


Figure (4.41): bacteria activity of E.Coli

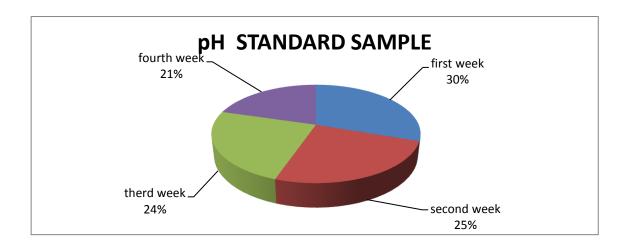


Figure (4.42): Statistical bacteria activity E-Coli results of pH for the standard sample.

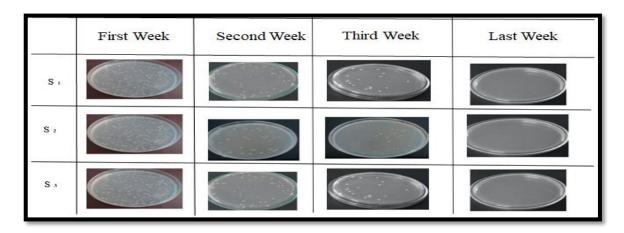


Figure (4.43): Antibacterial activities of the Azadirchta Indica seeds (Neem seeds) against of E-Coli.

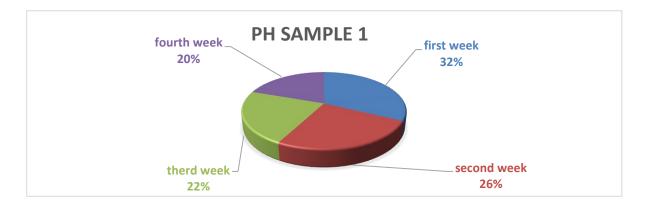


Figure (4.44): Statistical bacteria activity E-Coli results of pH for Azadirchta

Indica seeds dissolved by ethanol.

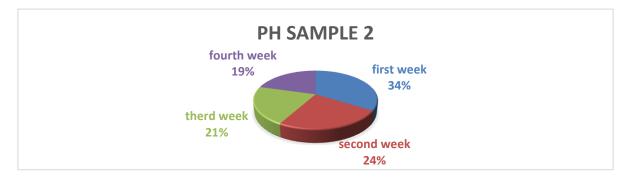


Figure (4.45): Statistical bacteria activity E-Coli results of pH for Azadirchta Indica seeds dissolved by methanol.

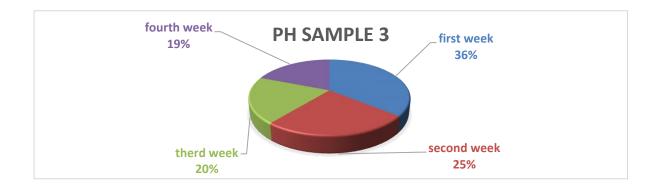


Figure (4.46): Statistical bacteria activity E-Coli results of pH for Azadircht Indica seeds dissolved by water.

|      | First Week | Second Week | Third Week | Last Week  |
|------|------------|-------------|------------|------------|
| S 21 | $\bigcirc$ | $\bigcirc$  | $\bigcirc$ |            |
| S 22 | $\bigcirc$ | $\bigcirc$  |            | $\bigcirc$ |
| S 23 | $\bigcirc$ | $\bigcirc$  | $\bigcirc$ | $\bigcirc$ |

Figure (4.47): Antibacterial activities of the Trigonella Foenum Graecum seeds against of E-Coli.

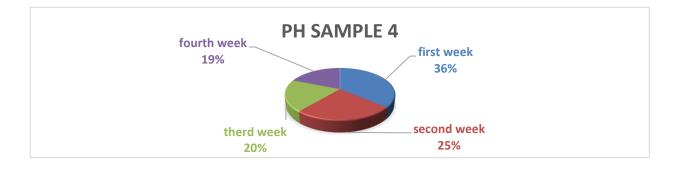


Figure (4.48): Statistical bacteria activity E-Coli results of pH for Trigonella Foenum Graecum seeds dissolved by ethanol

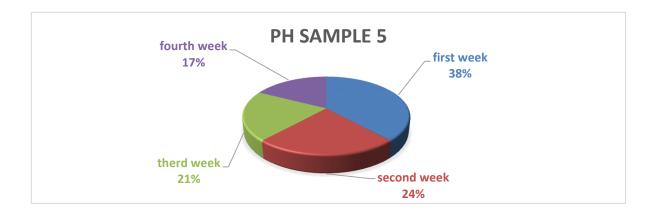


Figure (4.49): Statistical bacteria activity E-Coli results of pH for Trigonella Foenum Graecum seeds dissolved by methanol.

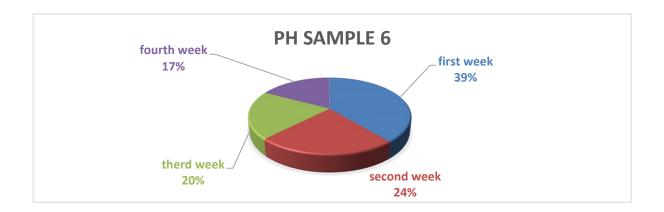
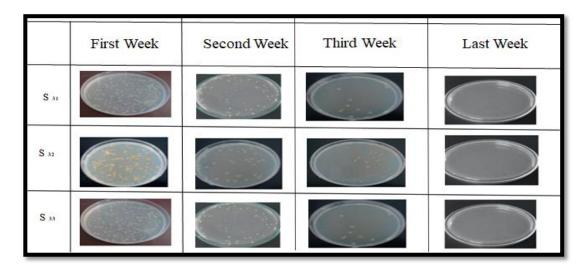


Figure (4-50): Statistical bacteria activity E-Coli results of pH for Trigonella Foenum Graecum seeds dissolved by water



. Figure (4.51): Antibacterial activities of the negella sativa seeds against of E-Coli.

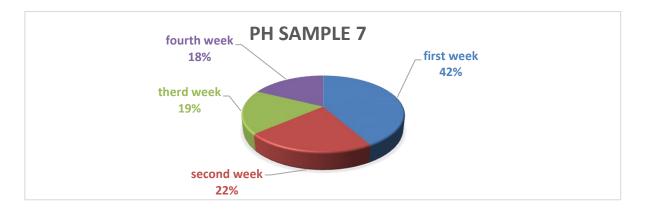


Figure (4.52): Statistical bacteria activity E-Coli results of pH for Negella sativa seeds dissolved by ethanol.

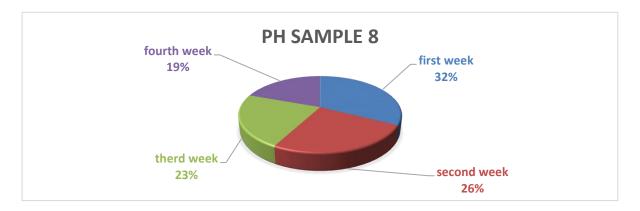


Figure (4.53): Statistical bacteria activity E-Coli results of pH for Negella sativa seeds dissolved by methanol.

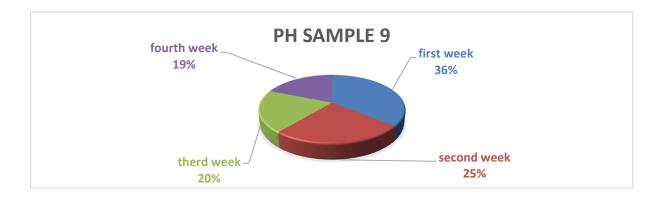


Figure (4.54): Statistical bacteria activity E-Coli results of pH for Negella sativa seeds dissolved by water.

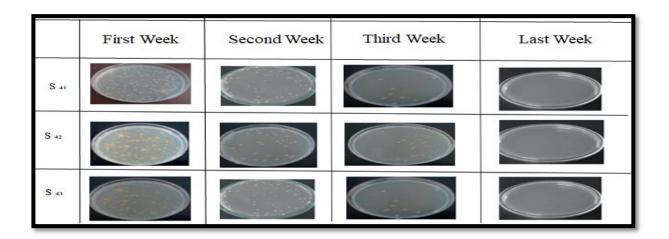


Figure (4.55): Antibacterial activities of the Elettaria Cardamomum seeds against of E-Coli.

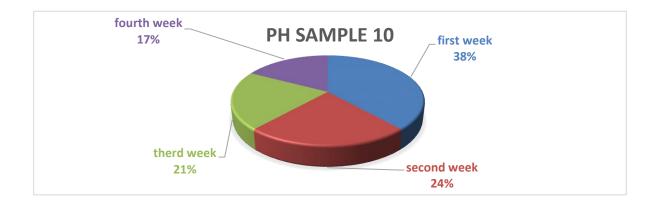


Figure (4.56): Statistical bacteria activity E-Coli results of pH for Elettaria Cardamomum seeds dissolved by ethanol.

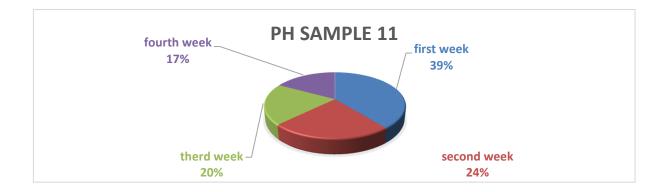
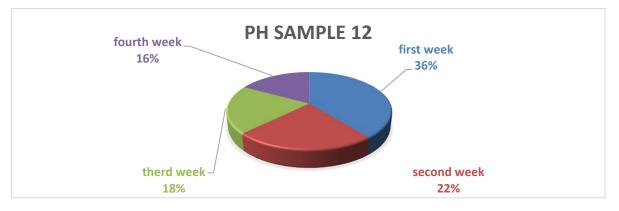


Figure (4.57): Statistical bacteria activity E-Coli results of PH for elettaria



cardamomum seeds dissolved by methanol.

Figure (4.58): Statistical bacteria activity E-Coli results of PH for elettaria cardamomum seeds dissolved by water

| Sam/no | Seed | Cha.co   | Solve    | (x <sub>s</sub> ) nm | n    | K(a.u | Е    | $\mu / 10^{-18}$ | $\sigma_0/10^{14}$ | $\sigma_E$ |
|--------|------|--|----------|----------------------|------|-------|------|------------------|--------------------|------------|
| 1      | Neem | O–H,& H–bond   | Water    | 100                  | 2.14 | 0.378 | 4.44 | 11.5             | 5.7                | 26.32      |
| 2      | Neem | O–H,& H–bond   | Methanol | 100                  | 2.14 | 0.179 | 4.56 | 11.16            | 5.8                | 21.73      |
| 3      | Neem | O–H,& H–bond   | Ethanol  | 100                  | 2.05 | 0.143 | 4.18 | 11.17            | 4.4                | 21.51      |
| 4      | T.F  | Fe <sub>2</sub> O <sub>3&amp;</sub> Fe <sub>2</sub> O <sub>4</sub> | Water    | 27.3                 | 1.94 | 1.45  | 1.66 | 25.19            | 17                 | 33.02      |
| 5      | T .F | Fe <sub>2</sub> O <sub>3&amp;</sub> Fe <sub>2</sub> O <sub>4</sub> | Methanol | 27.3                 | 2    | 0.899 | 3.19 | 13.93            | 17.9               | 29.16      |
| 6      | F.T  | Fe <sub>2</sub> O <sub>3&amp;</sub> Fe <sub>2</sub> O <sub>4</sub> | Ethanol  | 27.3                 | 2.05 | 0.69  | 1.66 | 14.64            | 17.4               | 27.16      |
| 7      | N .S | C-Br & N-O   | Water    | 11.1                 | 2.14 | 0.378 | 4.44 | 11.46            | 5.7                | 26.32      |
| 8      | N. S | C-Br & N-O   | Methanol | 11.1                 | 2.14 | 0.179 | 4.55 | 11.18            | 5.2                | 21.73      |
| 9      | N. S | C-Br & N-O   | Ethanol  | 11.1                 | 2.05 | 0.143 | 4.18 | 13.19            | 4.4                | 21.51      |
| 10     | E.C  | С-F & -С=С-Н-Н   | Water    | 48.4                 | 2.09 | 0.83  | 3.68 | 13.19            | 11.3               | 30.3       |
| 11     | E. C | С-F & -С=С-Н-Н   | Methanol | 48.4                 | 2.11 | 0.89  | 3.76 | 13.16            | 11.3               | 35.05      |
| 12     | E. C | С-F & -С=С-Н-Н   | Ethanol  | 48.4                 | 2.11 | 1.2   | 3.01 | 16.32            | 11.2               | 37.37      |

Table (4.10): The relations between seeds characteristic bonds, crystal size and electric and magnetic properties:

## **Chapter five**

### **DISCUSSION, CONCLUSIONS AND RECOMMENDATION**

#### **5.1. Introduction**

This chapter is concerned with discussion, conclusion and recommendation.

#### 5.2. Discussion

In this work four different seeds (azadirchta indica (neem seeds), trigonella foenum graecum, nigella sativa and elettaria cardamomum) were dissolved in three different solvents (water, methanol and ethanol). Then they were added to water in which coli are living.

The XRD results for all samples powders indicates that all samples are in the form of nano particles, as shown in figures (4.1), (4.2), (4.3) and (4.4) and tables (4.1),(4.2), (4.3) and (4.4). The optical properties of the 12 samples (four seeds × three solvents) were studied using (UV) spectrometer. To determine the characteristic compounds which exist only in one of the seeds type, the four seeds bonds were determined using FTIR spectrometer. It shows that the characteristic bonds for Azadirchta Indica seed are O–H, & H–bond, for trigonella foenum graecum are Fe<sub>2</sub>o<sub>3</sub> & Fe<sub>2</sub>o<sub>4</sub>, for Negella sativa are C-Br & N-O and for elettaria cardamomum are C-F & -C=C-H-H. The UV- spectra to azadirchta indica seeds (Neem seeds) which determines the absorption, extension coefficient, refractive index, optical and electrical conductivities are shown in figures [(4.9), (4.13), (4.14), (4.25), and (4.16)]. The UV- spectra to Trigonella Foenum Graecum seeds which determines the absorption, extension coefficient, optical and electrical conductivities are shown in figures [(4.21), (4.22), (4.23), and (4.24)]. The UV- Spectra to Negella sativa seeds which determines the

absorption, extension coefficient, refractive index, optical and electrical conductivities are shown in figures [(4.25), (4.29), (4.30), (4.31), and (4.32)]. The UV- Spectra to Elettaria Cardamomum seeds which determines the absorption, extension coefficient, refractive index, optical and electrical conductivities are shown in figures [(4.33), (4.37), (4.38), (4.39), and (4.40)]. Using some theoretical relations the electric permittivity and magnetic permeability were found and tabulated together with conductivities in table (4.10). The results obtained indicate that all these parameters changes with the solvent type and seed type. The fact that these quantities changes with solvent types which changes the nano size (as pointed out by Asia [79]) indicates that the change of nano size changes these physical quantities. According to Asia paper the methanol crystal nano size and absorption coefficient are greater than that of ethanol. This conforms with our absorption coefficient result. The results show that the electric permittivity for the four seeds decreases for methanol, water, and ethanol solvents respectively. The electric conductivity for Azadirchta Indica seeds (Neem seeds) are (26.32, 21.73, and 21.51), Trigonella Foenum Graecum seeds are (33.2, 29.16 and 27.16), Negella sativa seeds are (26.32, 21.73 and 21.51) and Elettaria Cardamomum seeds are (30.3, 35.05 and 37.37) for water, methanol and ethanol respectively. For the magnetic permittivity it is very interesting to observe that the existence of Fe in fenugreek which has high magnetic moment increases it considerably where it attains maximum value of  $25.19 \times 10^{-18}$  for water (as shown by tables (4.10). The PH of water of for the 12 sample beside the control in which no seeds were added shows very intersecting results as shown by table (4.9) and figures (4.42), (4.44), (4.45),(4.46),(4.48),(4.49),(4.50),(4.52),(4.53),(4.54),(4.56),(4.57),(4.58) and (4.59). The results show that the PH decreases in general for Ethanol, Methanol and Water respectively. It also decreases with time weakly where it attains maximum value in the first week than take minimum value in the fourth week.

## 5.3 Conclusions

The change of chemical bonds and crystal size of the four nano powder seeds (Azadirchta Indica (Neem seeds), Trigonella Foenum Graecum, Negella sativa and Elettaria Cardamomum) due to the change of solvents affect and change electric permittivity and conductivity beside the magnetic permittivity. The PH and E.coli which reflect deionization and change of internal thermodynamic energy changes also with chemical bonds and solvents beside time.

### **5.4 Recommendation**

- **I.** The change of nano size for different solvents needs to be investigated scanning electron microscope (SEM).
- **II.** The determination of trace elements in seeds is very important using X-Ray fluorescence technique or laser break down technique.
- III. The physical properties of other seeds used as food supplements needs also to be studied.
- IV. The link between the changes of water physical properties a due to addition of these seeds and the effect of these changes on the human health is very important.

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# **Appendix: publishing arising out of this thesis:**

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